

REMARKS

This is in response to the Office Action that was mailed on March 1, 2004.

The Examiner is respectfully requested to indicate acceptance of the drawings filed with this application.

A new title of the invention has been supplied, as requested by the Examiner.

Claims 1-9 were rejected under the second paragraph of 35 U.S.C. §112. The rejection queried the units for the penetration values and hardness values recited in the claims, and requested documentation of the testing standards JIS K 2220 and JIS K 6253 recited in the claims.

Copies of JIS K 2220 (1993) and JIS K 6253 (1997) are enclosed herewith. Also enclosed is a copy of ASTM D 1403-83, which corresponds to JIS K 2220.

The category of "elastomers" includes both rubbers and gels. The harness of rubbers is measured by using a Durometer or hardness tester defined in JIS K 6253. In the present invention, Durometer A testing is used in the Examples.

Gels are often too soft, due to their low crosslinking density, to have their hardness measured by a Durometer A tester.

The hardness of gels is measured by using a penetrometer as in JIS K 2220 (or ASTM D 1403).

Neither the penetration values nor the hardness values provided, respectively, in accordance with JIS K 2220 and JIS K 6253, have units.

As is disclosed in the present specification, the invention here provides for leads and bonding wires in a semiconductor pressure sensor being covered and protected with an electrically insulating fluorochemical gel material. The fluorochemical gel material used in this invention has a degree of saturation swelling in gasoline at 23°C of up to 7% by weight, a glass transition temperature of up to -45°C, and a penetration of 30-60 as measured by the consistency test of JIS K 2220 using a  $\frac{1}{4}$  cone. As long as the penetration of the gel material is within the above-defined range, bubble formation in the gel material is prevented due to shortage of adhesive strength at a negative pressure or a low temperature. Bubble formation under pressure cycling at a low temperature is prevented. Since no bubbles generate in proximity to the portions covered with the fluorochemical gel materials - specifically in proximity to the sensitive portion of the sensor chip, the bonding wires and the connections between the bonding wires and the sensor chip or leads - the problem of degradation of insulating function or breakage of bonding wires caused by bubble formation is overcome by this invention.

A second aspect of this invention adds the requirement that the hardness of the fluorochemical rubber material be limited to the range of 5 to 35. Materials with hardnesses of less than 5 provide insufficient bubble restraining effect at high temperatures and negative pressures. Materials with hardnesses of more than 35 may fail to respond appropriately to a change of pressure or temperature, and accordingly may separate from the surface to be covered or cause breakage to the bonding wires.

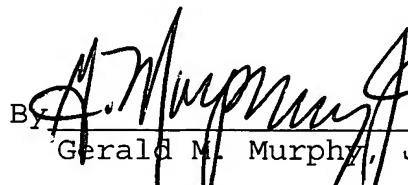
It is respectfully submitted that the claims herein satisfy the requirements of the statute. Accordingly, the rejection of record should be withdrawn, and this application should be passed to issue.

Should there be any outstanding matters that need to be resolved in the present application, the Examiner is respectfully requested to contact Rick Gallagher (Reg. No. 28,781) at the telephone number of the undersigned below, to conduct an interview in an effort to expedite prosecution in connection with the present application.

If necessary, the Commissioner is hereby authorized in this, concurrent, and future replies, to charge payment or credit any overpayment to Deposit Account No. 02-2448 for any additional fees required under 37 C.F.R. §§ 1.16 or 1.17; particularly, extension of time fees.

Respectfully submitted,

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Attachment(s): JIS K 2220 (1993);  
JIS K 6253 (1997); and  
ASTM D 1403-83

# JIS

**JAPANESE INDUSTRIAL STANDARD**

**Lubricating grease**

 **JIS K 2220-1993**

**Translated and Published**

**by**

**Japanese Standards Association**

**In the event of any doubt arising,  
the original Standard in Japanese is to be final authority**

Errata for JIS (English edition) are printed in *Standardization Journal*, published monthly by the Japanese Standards Association.

Errata will be provided upon request, please contact:  
**Business Department,**  
**Japanese Standards Association**  
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Errata are also provided to subscribers of JIS (English edition) in *Monthly Information*.

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1. Scope This Japanese Industrial Standard specifies the lubricating grease (hereafter referred to as "grease") to be used mainly as the lubricants for various kinds of machine parts.

Remarks 1. The standards cited in this Standard are shown in Attached Table 1.

2. Corresponding International Standards to this Standard are shown as follows.

ISO 2137: 1985 Petroleum products - Lubricating grease and petrolatum - Determination of cone penetration

ISO 2176: 1972 Petroleum products - Lubricating grease - Determination of dropping point

3. The units and numerical values given in { } in this Standard are based on the traditional units and are standard values.

Further, those traditional units and numerical values shall be informative reference values on and after April 1st, 1995.

2. Definitions The principal terms used in this Standard shall be as defined below.

- (1) grease A solid or semisolid matter produced by dispersing a thickening agent in a lubricating oil. It may contain other ingredients to impart special properties.
- (2) gear compound A matter produced by dissolving or dispersing a viscous substance such as asphalt and others in mineral oil. It is used principally for the lubricants for open gears. It may contain other ingredients to impart special properties.
- (3) base oil Lubricating oil being a raw material for grease. The base oil is roughly classified into refined mineral oil, synthetic lubricating oil, and their mixed oil.
- (4) thickening agent Substance, dispersing in lubricating oil in colloidal state, to render it semisolid or solid. Thickening agents are classified principally into two types; metallic soap type and non-soap type. The former is represented by metallic soaps of lithium, calcium, sodium and others, and the latter by inorganic compounds such as bentonite, silica gel and others, and organic compounds such as urea derivatives, phthalocyanine and others. Properties of grease greatly depend upon the type of thickening agent used.

(5) water resistance Resistance of the grease to moisture or water, it is to say the property of grease to withstand without being influenced when contacted with moisture or water in practical use.

(6) mechanical stability Resistance of the grease to the change in hardness when subjected to a mechanical shearing force. It is also called shearing stability.

(7) property of delivery under pressure The fluid properties of grease when it is fed under pressure through piping, nozzle and accessories of a lubricating system.

(8) load carrying capacity Maximum load or pressure under which lubrication can continue without causing defects such as seizure or fusion on the bearing or the sliding surface when a lubricant is used under the specified conditions.

(9) cone penetration Depth expressed in 10 times millimetre to which a specified cone penetrates into the sample grease vertically in the specified time. It indicates the consistency of a grease.

(10) working To subject grease to the shearing action of the specified grease worker.

(11) worked penetration Cone penetration of the sample grease, which have been maintained at 25°C in the specified worker, measured immediately after working 60 double strokes.

(12) unworked penetration Cone penetration, at 25°C, of the sample grease which has been transferred, without disturbance as possible, from sample container to the specified worker or the vessel having the same dimensions.

(13) prolonged worked penetration Cone penetration, obtained immediately after 60 double strokes, of the sample grease which has been worked in the specified worker to the specified number of strokes and then kept at 25°C.

(14) undisturbed penetration Cone penetration of the sample at 25°C, as it is without being stirred, after stored for a specified period in the specified container.

(15) block penetration Cone penetration of the sample grease, measured at 25°C, of which consistency is enough to keep shape, after it was cut into the specified dimensions.

(16) quarter and half cone penetrations The value defined as the depth, expressed in 10 times millimetre, to which the specified cones, miniaturized to a quarter and a half size of the standard cone, penetrate into the sample grease in the specified time.

(17) numbers of cone penetration The number classified according to the ranges of worked penetrations of greases, which are as enumerated in Table 1.

Table 1. Numbers of cone penetration

Number of cone penetration	Range of worked penetration
No. 000	445 to 475
No. 00	400 to 430
No. 0	355 to 385
No. 1	310 to 340
No. 2	265 to 295
No. 3	220 to 250
No. 4	175 to 205
No. 5	130 to 160
No. 6	85 to 115

(18) dropping point Dropping point is defined as the temperature at which the first drop of the grease falls, when a sample is heated in the specified apparatus under the specified conditions and beginning to change its state from semisolid to liquid state.

(19) worked stability After working grease sample one hundred thousand times in the specified grease worker, hold at 25°C, then immediately after working 60 double strokes, measure the cone penetration to be taken as worked stability.

(20) water washout resistance The loss of a sample measured 1 h after a specified ball bearing packed with the sample is revolved at a specified number of revolutions ( $600 \pm 30/\text{min}$ ), and the distilled water kept at a specified temperature is sprayed onto the housing of the ball bearing at a specified rate ( $5 \pm 0.5 \text{ ml/s}$ ).

(21) low temperature torque Low temperature torque is defined as the force enough to restrain the turning of the outer ring of the specified open type ball bearing, in which the grease sample is packed and of which inner ring is turned at the specified rate of revolution (one turn per minute) at the specified low temperature. It is expressed in two classified torques described below.

(a) Starting torque The largest torque obtained at the start of revolution.

(b) Revolving torque The averaged torque obtained after revolutions for the specified time.

(22) apparent viscosity Apparent viscosity is defined as the ratio of shear stress to shear rate calculated from Poiseuille's formula. Since grease is non-Newtonian fluid, the ratio will vary with the shear rate.

(23) shear rate Rate of a series of adjacent layers of grease to move each other.

3. Classification Grease shall be classified into 7 classes according to the use, and shall be further subdivided as shown in Table 2 according to grades (components and performance), and the number of cone penetration (worked penetration range or viscosity range).

Table 2. Classification

Classification			Applicable temperature range °C	Applicability to using condition				Example of application		
Class according to use	Grade	No. of cone penetration		Load		Contact with water				
				Light	Heavy	Impact				
Grease for general purpose	Grade 1	No.1, No.2, No.3, No.4	-10 to 60	Applicable	Inapplicable	Inapplicable	Applicable	For general light load		
	Grade 2	No.2, No.3	-10 to 100	Applicable	Inapplicable	Inapplicable	Inapplicable	For general medium load		
Grease for rolling bearing	Grade 1	No.1, No.2, No.3	-20 to 100	Applicable	Inapplicable	Inapplicable	Applicable	For general purpose		
	Grade 2	No.0, No.1, No.2	-40 to 80	Applicable	Inapplicable	Inapplicable	Applicable	For low temperature		
Grade 3	No.1, No.2, No.3	-30 to 130	Applicable	Inapplicable	Inapplicable	Inapplicable	Applicable	For wide temperature range		
	Chassis grease for automobile	No.00, No.0, No.1, No.2	-10 to 60	Applicable	Applicable	Applicable	Applicable	For chassis of automobile		
Wheel bearing grease for automobile	Grade 1	No.2, No.3	-20 to 120	Applicable	Inapplicable	Inapplicable	Applicable	For wheel bearing of automobile		

Table 2. (continued)

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Classification	Applicable temperature range °C	Informative reference					
		Applicability to using condition			Example of application		
		Load	Contact with water	Impact	Load	Contact with water	Impact
Class according to use	Grade	No. of cone penetration					
Grease for central lubricating system	Grade 1	No.0.0, No.0.1	-10 to 60	Applicable	Inapplicable	Inapplicable	Applicable
	Grade 2	No.0.0, No.1, No.2	-10 to 100	Applicable	Inapplicable	Inapplicable	Applicable
	Grade 3	No.0.0, No.1, No.2	-10 to 60	Applicable	Applicable	Applicable	Applicable
	Grade 4	No.0.0, No.1, No.2	-10 to 100	Applicable	Applicable	Applicable	Applicable
Grease for heavy load	Grade 1	No.0, No.1, No.2, No.3	-10 to 100	Applicable	Applicable	Applicable	Applicable
Gear compound	Grade 1	No.1(1), No.2(1), No.3(1)	-10 to 100	Applicable	Applicable	Applicable	Applicable

Note <sup>(1)</sup> The numbers given in this column are such those which are classified according to the range of viscosity.

Remarks

1. Grade 1 grease for general purpose principally consists of base oil and calcium soap, and has sufficient water resistance.
2. Grade 2 grease for general purpose principally consists of base oil and sodium soap, and has sufficient heat resistance.
3. Grade 1 grease for rolling bearing principally consists of base oil and thickening agent, and has sufficient mechanical stability, water resistance, and rust preventive property.
4. Grade 2 grease for rolling bearing principally consists of base oil and thickening agent, and has sufficient low temperature property, water resistance, mechanical stability, and rust preventive property.
5. Grade 3 grease for rolling bearing principally consists of base oil and thickening agent, and has sufficient low temperature property, heat resistance, mechanical stability, water resistance, and rust preventive property.
6. Grade 1 chassis grease for automobile principally consists of base oil and calcium soap, and has sufficient load carrying capacity and property of delivery under pressure.
7. Grade 1 wheel bearing grease for automobile principally consists of base oil and thickening agent, and sufficient heat resistance, water resistance, mechanical stability, and anti-leakage property.
8. Grade 1 grease for central lubricating system principally consists of base oil and calcium soap, and has sufficient property of delivery under pressure.
9. Grade 2 grease for central lubricating system principally consists of base oil and thickening agent, and has sufficient property of delivery under pressure, heat resistance, and mechanical stability.
10. Grade 3 grease for central lubricating system principally consists of base oil, calcium soap and extreme pressure additives, and has sufficient property of delivery under pressure and load carrying capacity.
11. Grade 4 grease for central lubricating system principally consists of base oil, thickening agent and extreme pressure additives, and has sufficient property of delivery under pressure, load carrying capacity and mechanical stability.

12. Grade 1 grease for heavy load principally consists of base oil, thickening agent and solid lubricants such as molybdenum disulfide or the like, and has sufficient load carrying capacity, mechanical stability, and heat resistance.

13. Grade 1 gear compound principally consists of base oil and asphalt.

#### 4. Quality and performance

##### 4.1 Grease for general purpose

4.1.1 Grade 1 grease for general purpose Grade 1 grease for general purpose, when subjected to the tests in accordance with 5., shall conform to requirements specified in Table 3.

Table 3. Grade 1 grease for general purpose

Item	Test method	No. of cone penetration	No. 1	No. 2	No. 3	No. 4
Worked penetration		5.3	310 to 340	265 to 295	220 to 250	175 to 205
Dropping point °C		5.4	80 min.	85 min.	85 min.	90 min.
Copper corrosion (room temp., 24 h)		5.5 (Method A)	No change to green or black colour on copper plate.			
Ash content mass %		5.10	3.0 max.	3.5 max.	4.0 max.	4.5 max.
Water washout resistance (38°C, 1 h) mass %		5.12	20 max.	20 max.	20 max.	20 max.
Water content mass %		5.18	2.0 max.	2.5 max.	2.5 max.	3.0 max.

Remarks: The base oil shall have kinematic viscosity (40°C) of 6.12 to 74.8 mm<sup>2</sup>/s{cSt}.

If necessary, kinematic viscosity shall additionally be stated in the test report.

4.1.2 Grade 2 grease for general purpose Grade 2 grease for general purpose, when subjected to the tests in accordance with 5., shall conform to the requirements specified in Table 4.

Table 4. Grade 2 grease for general purpose

Item	Test method	No. of cone penetration	No. 2	No. 3
Worked penetration		5.3	265 to 295	220 to 250
Dropping point °C		5.4	170 min.	170 min.
Copper corrosion (room temp., 24 h)		5.5 (Method A)	No change to green or black colour on copper plate.	
Evaporation loss (99°C, 22 h) mass %		5.6	2.0 max.	2.0 max.
Worked stability		5.11	375 max.	375 max.

Remarks: The base oil shall have kinematic viscosity (40°C) of 41.4 to 242 mm<sup>2</sup>/s {cSt}.

If necessary, kinematic viscosity shall additionally be stated in the test report.

#### 4.2 Grease for rolling bearing

4.2.1 Grade 1 grease for rolling bearing Grade 1 grease for rolling bearing, when subjected to the tests in accordance with 5., shall conform to the requirements specified in Table 5.

Table 5. Grade 1 grease for rolling bearing

Item	Test method	No. of cone penetration	No. 1	No. 2	No. 3
Worked penetration		5.3	310 to 340	265 to 295	220 to 250
Dropping point °C		5.4	170 min.	175 min.	175 min.
Copper corrosion (100°C, 24 h)		5.5 (Method B)	No change to green or black colour on copper plate.		
Evaporation loss (99°C, 22 h) mass %		5.6	2.0 max.	2.0 max.	2.0 max.
Oil separation percentage (100°C, 24 h) mass %		5.7	10 max.	5 max.	5 max.
Oxidation stability (99°C, 100 h) MPa{kgf/cm <sup>2</sup> }		5.8	0.069 {0.7} max.	0.069 {0.7} max.	0.069 {0.7} max.

Table 5. (continued)

Item	Test method		No. of cone penetration	No. 1	No. 2	No. 3
Number of deleterious particles particles/cm <sup>3</sup>	10 $\mu\text{m}$ min.	5.9	5000 max.	5000 max.	5000 max.	
	25 $\mu\text{m}$ min.		3000 max.	3000 max.	3000 max.	
	75 $\mu\text{m}$ min.		500 max.	500 max.	500 max.	
	125 $\mu\text{m}$ min.		0	0	0	
Worked stability		5.11	400 max.	375 max.	350 max.	
Water washout resistance (38°C, 1 h) mass %		5.12	10 max.	10 max.	10 max.	
Low temperature torque (-20°C) N·cm{gf·cm}	Starting torque	5.14	49{5000} max.	59{6000} max.	79{8000} max.	
	Revolving torque		25{2500} max.	29{3000} max.	39{4000} max.	
Humidity cabinet (14 days)		5.17	Class A	Class A	Class A	

Remarks: If necessary, kinematic viscosity of base oil and kind of thickening agent shall additionally be stated in the test report.

4.2.2 Grade 2 grease for rolling bearing Grade 2 grease for rolling bearing, when subjected to the tests in accordance with 5., shall conform to the requirements specified in Table 6.

Table 6. Grade 2 grease for rolling bearing

Item	Test method	No. of cone penetration	No. 0	No. 1	No. 2
Worked penetration		5.3	355 to 385	310 to 340	265 to 295
Dropping point °C		5.4	145 min.	150 min.	150 min.
Copper corrosion (100°C, 24 h)		5.5 (Method B)	No change to green or black colour on copper plate.		
Evaporation loss (99°C, 22 h) mass %		5.6	10.0 max.	10.0 max.	10.0 max.
Oil separation percentage (100°C, 24 h) mass %		5.7	-	12 max.	10 max.
Oxidation stability (99°C, 100 h) MPa{kgf/cm²}		5.8	0.069 {0.7} max.	0.069 {0.7} max.	0.069 {0.7} max.
Number of deleterious particles particles/cm³	10 µm min. 25 µm min. 75 µm min. 125 µm min.	5.9	5000 max. 3000 max. 500 max. 0	5000 max. 3000 max. 500 max. 0	5000 max. 3000 max. 500 max. 0
Worked stability		5.11	430 max.	400 max.	375 max.
Water washout resistance (38°C, 1 h) mass %		5.12	-	10 max.	10 max.
Low temperature Starting torque (-40°C) N·cm{gf·cm}	Starting torque Revolving torque	5.14	39{4000} max. 20{2000} max.	49{5000} max. 25{2500} max.	59{6000} max. 29{3000} max.
Humidity cabinet (14 days)		5.17	Class A	Class A	Class A

Remarks: If necessary, kinematic viscosity of base oil and kind of thickening agent shall additionally be stated in the test report.

4.2.3 Grade 3 grease for rolling bearing Grade 3 grease for rolling bearing, when subjected to the tests in accordance with 5., shall conform to the requirements specified in Table 7.

Table 7. Grade 3 grease for rolling bearing

Item	Test method	No. of cone penetration	No. 1	No. 2	No. 3
Worked penetration		5.3	310 to 340	265 to 295	220 to 250
Dropping point	°C	5.4	180 min.	185 min.	185 min.
Copper corrosion (100°C, 24 h)		5.5 (Method B)	No change to green or black colour on copper plate.		
Evaporation loss mass %	99°C, 22 h	5.6	1.5 max.	1.5 max.	1.5 max.
	130°C, 22 h		5.0 max.	5.0 max.	5.0 max.
Oil separation percentage mass %	100°C, 24 h	5.7	10 max.	5 max.	5 max.
	130°C, 24 h		12 max.	8 max.	8 max.
Oxidation stability (99°C, 100 h) MPa{kgf/cm <sup>2</sup> }		5.8	0.049 {0.5} max.	0.049 {0.5} max.	0.049 {0.5} max.
Number of deleterious particles particles/cm <sup>3</sup>	10 µm min.	5.9	5000 max.	5000 max.	5000 max.
	25 µm min.		3000 max.	3000 max.	3000 max.
	75 µm min.		500 max.	500 max.	500 max.
	125 µm min.		0	0	0
Worked stability		5.11	400 max.	375 max.	350 max.
Water washout resistance (38°C, 1 h) mass %		5.12	10 max.	10 max.	10 max.
Low temperature Starting torque (-30°C) torque N·cm{gf·cm}	Revolving torque	5.14	49{5000} max.	59{6000} max.	79{8000} max.
			25{2500} max.	29{3000} max.	39{4000} max.
Humidity cabinet (14 days)		5.17	Class A	Class A	Class A

Remarks: If necessary, kinematic viscosity of base oil and kind of thickening agent shall additionally be stated in the test report.

4.3 Chassis grease for automobile Grade 1 chassis grease for automobile, when subjected to the tests specified in 5., shall conform to the requirements specified in Table 8.

Table 8. Grade 1 chassis grease for automobile

Item	Test method	No. of cone penetration	No. 00	No. 0	No. 1	No. 2
Worked penetration		5.3	400 to 430	355 to 385	310 to 340	265 to 295
Dropping point °C		5.4	80 min.	85 min.	90 min.	90 min.
Copper corrosion (room temp., 24 h)		5.5 (Method A)	No change to green or black colour on copper plate.			
Water washout resistance (38°C, 1 h) mass %		5.12	-	-	20 max.	10 max.
Apparent viscosity Pa·s{P} (-10°C, shear rate 10 s <sup>-1</sup> )		5.15	100{1000} max.	200{2000} max.	-	-
Load carrying capacity by Timken machine OK value kg		5.16	4.08 min.	4.08 min.	4.08 min.	4.08 min.
Humidity cabinet (14 days)		5.17	-	-	Class A	Class A
Water content mass %		5.18	2.0 max.	2.0 max.	2.0 max.	2.0 max.

Remarks: If necessary, kinematic viscosity of base oil shall additionally be stated in the test report.

4.4 Wheel bearing grease for automobile Grade 1 wheel bearing grease for automobile, when subjected to the tests in accordance with 5., shall conform to the requirements specified in Table 9.

Table 9. Grade 1 wheel bearing grease for automobile

Item	Test method	No. of cone penetration	No. 2	No. 3
Worked penetration		5.3	265 to 295	220 to 250
Dropping point	°C	5.4	175 min.	175 min.
Copper corrosion (100°C, 24 h)		5.5 (Method B)	No change to green or black colour on copper plate.	
Evaporation loss (99°C, 22 h)	mass %	5.6	2.0 max.	2.0 max.
Oil separation percentage (100°C, 24 h)	mass %	5.7	5 max.	5 max.
Oxidation stability (99°C, 100 h)	MPa{kgf/cm <sup>2</sup> }	5.8	0.069 {0.7} max.	0.069 {0.7} max.
Number of deleterious particles particles/cm <sup>3</sup>		5.9	5000 max. 3000 max. 500 max. 0	5000 max. 3000 max. 500 max. 0
Worked stability		5.11	375 max.	375 max.
Water washout resistance (79°C, 1 h)	mass %	5.12	10 max.	10 max.
Leakage tendency (104°C, 6 h)	g	5.13	10 max.	10 max.
Low temperature Starting torque -20°C torque	N·cm{gf·cm}	5.14	79{8000} max. 39{4000} max.	99{10000} max. 49{5000} max.
Humidity cabinet (14 days)		5.17	Class A	Class A

Remarks: If necessary, kinematic viscosity of base oil and kind of thickening agent shall additionally be stated in the test report.

#### 4.5 Grease for central lubricating system

4.5.1 Grade 1 grease for central lubricating system Grade 1 grease for central lubricating system, when subjected to the tests in accordance with 5., shall conform to the requirements specified in Table 10.

Table 10. Grade 1 grease for central lubricating system

Item	Test method	No. of cone penetration	No. 00	No. 0	No. 1
Worked penetration		5.3	400 to 430	355 to 385	310 to 340
Dropping point °C		5.4	80 min.	85 min.	90 min.
Copper corrosion (room temp., 24 h)		5.5 (Method A)	No change to green or black colour on copper plate.		
Apparent viscosity Pa·s{P} (-10°C, shear rate 10 s <sup>-1</sup> )		5.15	150{1500} max.	200{2000} max.	400{4000} max.
Load carrying capacity by Timken machine OK value kg		5.16	2.72 min.	2.72 min.	2.72 min.
Water content mass %		5.18	2.0 max.	2.0 max.	2.0 max.

Remarks: If necessary, kinematic viscosity of base oil shall additionally be stated in the test report.

4.5.2 Grade 2 grease for central lubricating system Grade 2 grease for central lubricating system, when subjected to the tests in accordance with 5., shall conform to the requirements specified in Table 11.

Table 11. Grade 2 grease for central lubricating system

Item	Test method	No. of cone penetration	No. 0	No. 1	No. 2
Worked penetration		5.3	355 to 385	310 to 340	265 to 295
Dropping point	°C	5.4	170 min.	170 min.	170 min.
Copper corrosion (100°C, 24 h)		5.5 (Method B)	No change to green or black colour on copper plate.		
Evaporation loss (99°C, 22 h)	mass %	5.6	2.0 max.	2.0 max.	2.0 max.
Oil separation percentage (100°C, 24 h)	mass %	5.7	-	10 max.	5 max.
Number of deleterious particles/cm <sup>3</sup>	25 µm min. 75 µm min. 125 µm min.	5.9	3000 max. 500 max. 0	3000 max. 500 max. 0	3000 max. 500 max. 0
Worked stability		5.11	430 max.	400 max.	375 max.
Water washout resistance (38°C, 1 h)	mass %	5.12	-	20 max.	10 max.
Apparent viscosity Pa·s{P} (-10°C, shear rate 10 s <sup>-1</sup> )		5.15	150{1500} max.	250{2500} max.	500{5000} max.
Load carrying capacity by Timken machine OK value kg		5.16	2.72 min.	2.72 min.	2.72 min.
Humidity cabinet (14 days)		5.17	Class A	Class A	Class A

Remarks: If necessary, kinematic viscosity of base oil and kind of thickening agent shall additionally be stated in the test report.

4.5.3 Grade 3 grease for central lubricating system Grade 3 grease for central lubricating system, when subjected to the tests in accordance with 5., shall conform to the requirements specified in Table 12.

Table 12. Grade 3 grease for central lubricating system

Item	Test method	No. of cone penetration	No. 0	No. 1	No. 2
Worked penetration		5.3	355 to 385	310 to 340	265 to 295
Dropping point	°C	5.4	80 min.	85 min.	90 min.
Copper corrosion (room temp., 24 h)		5.5 (Method A)	No change to green or black colour on copper plate.		
Apparent viscosity Pa·s{P} (-10°C, shear rate 10 s <sup>-1</sup> )		5.15	200{2000} max.	400{4000} max.	700{7000} max.
Load carrying capacity by Timken machine OK value kg		5.16	9.53 min.	9.53 min.	9.53 min.
Water content	mass %	5.18	2.0 max.	2.0 max.	2.0 max.

Remarks: If necessary, kinematic viscosity of base oil shall additionally be stated in the test report.

4.5.4 Grade 4 grease for central lubricating system Grade 4 grease for central lubricating system, when subjected to the tests in accordance with 5., shall conform to the requirements specified in Table 13.

Table 13. Grade 4 grease for central lubricating system

Item	Test method	No. of cone penetration	No. 0	No. 1	No. 2
Worked penetration		5.3	355 to 385	310 to 340	265 to 295
Dropping point	°C	5.4	170 min.	170 min.	170 min.
Copper corrosion (100°C, 24 h)		5.5 (Method B)	No change to green or black colour on copper plate.		
Evaporation loss (99°C, 22 h)	mass %	5.6	2.0 max.	2.0 max.	2.0 max.
Oil separation percentage (100°C, 24 h)	mass %	5.7	-	10 max.	5 max.
Number of deleterious particles/cm <sup>3</sup>	25 µm min. 75 µm min. 125 µm min.	5.9	3000 max. 500 max. 0	3000 max. 500 max. 0	3000 max. 500 max. 0
Worked stability		5.11	430 max.	400 max.	375 max.
Water washout resistance (38°C, 1 h)	mass %	5.12	-	20 max.	10 max.
Apparent viscosity Pa·s{P} (-10°C, shear rate 10 s <sup>-1</sup> )		5.15	150{1500} max.	250{2500} max.	500{5000} max.
Load carrying capacity by Timken machine OK value kg		5.16	10.9 min.	10.9 min.	10.9 min.
Humidity cabinet (14 days)		5.17	Class A	Class A	Class A

Remarks: If necessary, kinematic viscosity of base oil and kind of thickening agent shall additionally be stated in the test report.

4.6 Grease for heavy load Grade 1 grease for heavy load, when subjected to the tests in accordance with 5., shall conform to the requirements specified in Table 14.

Table 14. Grade 1 grease for heavy load

Item	Test method	No. of cone penetration	No. 0	No. 1	No. 2	No. 3
Worked penetration		5.3	355 to 385	310 to 340	265 to 295	220 to 250
Dropping point	°C	5.4	170 min.	170 min.	170 min.	175 min.
Copper corrosion (100°C, 24 h)		5.5 (Method B)	No change to green or black colour on copper plate.			
Evaporation loss (99°C, 22 h)	mass %	5.6	2.0 max.	2.0 max.	2.0 max.	2.0 max.
Oil separation percentage (100°C, 24 h)	mass %	5.7	-	10 max.	5 max.	5 max.
Worked stability		5.11	430 max.	400 max.	375 max.	350 max.
Water washout resistance (38°C, 1 h)	mass %	5.12	-	20 max.	10 max.	10 max.
Load carrying capacity by Timken machine		5.16	15.9 min.	15.9 min.	15.9 min.	15.9 min.
OK value kg						
Humidity cabinet (14 days)		5.17	Class A	Class A	Class A	Class A

Remarks: If necessary, kinematic viscosity of base oil and kinds of thickening agent and solid lubricant shall additionally be stated in the test report.

4.7 Gear compound Grade 1 gear compound, when subjected to the tests in accordance with 5., shall conform to the requirements specified in Table 15.

Table 15. Grade 1 gear compound

Item	Test method	No. of cone penetration	No. 1	No. 2	No. 3
Kinematic viscosity mm <sup>2</sup> /s{cSt}(100°C)		5.19	50 to 120	121 to 360	361 to 720
Flash point (C.O.C.) °C		5.20	150 min.	150 min.	150 min.
Load carrying capacity by four-ball machine MPa{kgf/cm <sup>2</sup> }		5.21	0.2{2.0} min.	0.2{2.0} min.	-
Copper corrosion (100°C, 24 h)		5.5 (Method B)	No change to green or black colour on copper plate.		
Ash content	mass %	5.10	4.0 max.	4.0 max.	4.0 max.

Remarks: The number of cone penetration has been classified according to the range of viscosity.

### 5. Test methods

5.1 Sampling method The grease shall be sampled in accordance with JIS K 2251.

5.2 Testing apparatus in general The apparatus shall have no potential danger, be durable enough, correct in shape, and satisfactory in assembly, and shall not easily get out of order mechanically and electrically, under the condition of ordinary use.

Insulation resistance of electric circuit shall be 5MΩ or over when tested with a 500 V insulation resistance tester between the live part and the frame. However, in the case where heater circuit is contained, 0.5 MΩ or over may be acceptable.

### 5.3 Test method for cone penetration

5.3.1 Summary of the test method Let a cone penetrate into a sample for 5 s, and measure the following penetrations.

- (1) Worked penetration After keeping a sample in the grease worker at 25°C, immediately after working 60 double strokes, measure the penetration.
- (2) Unworked penetration Transfer a sample into the grease worker without disturbance as possible, measure the penetration after keeping the sample at 25°C.
- (3) Prolonged worked penetration After working the sample for a specified strokes in grease worker, measure worked penetration specified in (1).

- (4) Undisturbed penetration After storing or leaving the sample contained in specified container as it is for a fixed period, keep the sample at 25°C, and measure the penetration.
- (5) Block penetration After keeping the sample which has been cut with a cutter at 25°C, measure the penetration on the cutting surface.

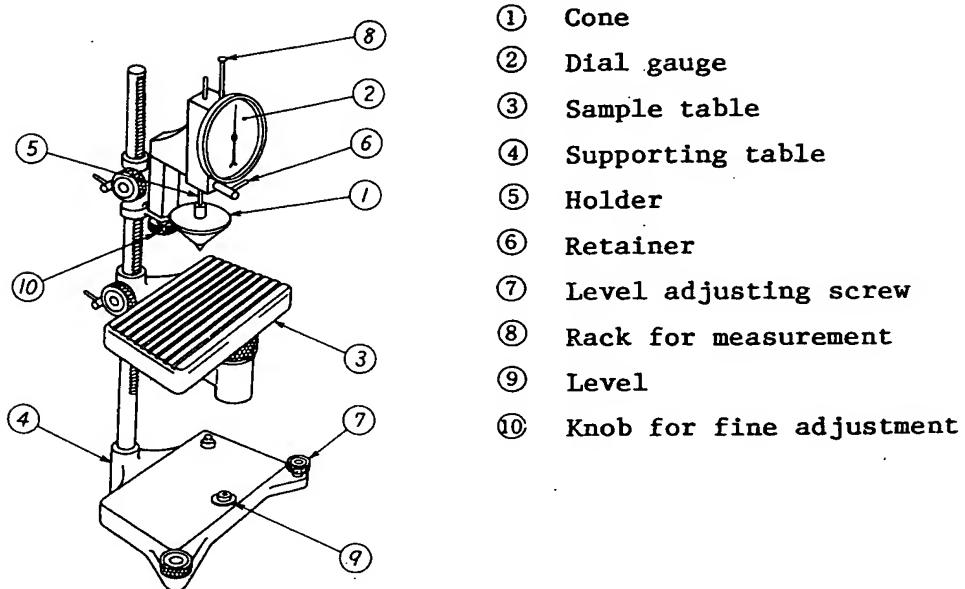
5.3.2 Tester for cone penetration The tester for cone penetration shall be composed of the following (1) to (9) items.

- (1) Penetrometer Penetrometer shall be of the construction shown in Fig. 1, equipped with such mechanism that by penetrating the specified cone (A or B) into a sample perpendicularly, the depth can be measured to the nearest 0.1 mm, and shall consist of falling down apparatus with retainer, dial gauge, sample table, supporting table with level adjusting screw, and others. In this case, sample table and supporting table may be used commonly.

Further, it shall be equipped with a minute vertical movement mechanism which enables fine adjustment when testing, of the distance between the sample surface on the sample table and the top of the cone attached to the holder.

- (a) Falling down apparatus It shall be so constructed that only during the automatical or manual acting of retainer, the cone can penetrate into the sample perpendicularly with the holder. Further, it shall have least frictional resistance as possible to the falling, and the falling distance of 50 mm or over.
- (b) Holder This is to hold the cone and shall weigh  $47.50 \pm 0.02$  g in mass.
- (c) Dial gauge The dial gauge shall have such mechanism that a moving distance is indicated on a scale when the rack for measurement is pressed down. The moving distance shall be read to the nearest 0.1 mm.
- (d) Supporting table The supporting table shall consist of supporting shaft for holding the dial gauge and the sample table, the table with level adjusting screw and others.

Fig. 1. Penetrometer (An example)



- (2) Cone A<sup>(2)</sup> This cone shall have the shape, dimensions and mass as shown in Fig. 2, and its outside surface shall be finished smooth.
- (3) Cone B<sup>(2)</sup> This cone shall have the shape, dimensions and mass as shown in Fig. 3, and its outside surface shall be finished smooth.
- (4) Grease worker<sup>(3)</sup> This shall have the shape and dimensions as shown in Fig. 4, and is capable of moving up and down the perforated plate attached to the top of its sliding spindle.

Notes (2) In the case of measuring the cone penetration of sample having the cone penetration of 400 or under, cone A (made of brass) or cone B (made of light metals) shall be used. However, in the case of sample having cone penetration exceeding 400, cone B shall be used.

(3) In the case of measuring the cone penetration of sample having the cone penetration exceeding 400, the over-flow ring shown in Fig. 5 shall be attached.

Remarks: When a water bath is used for the sample of which undisturbed penetration and unworked penetration are to be measured, the preparation shall be made so that a suitable cover may be used for preventing the surface of the sample from water entering and for maintaining the air temperature on the water surface at 25°C.

- (5) Over-flow ring This shall have the shape and dimensions as shown in Fig. 5, and be attached to the grease worker.

Fig. 2. Cone A

102.5  $\pm$  0.05 g in mass

Unit: mm

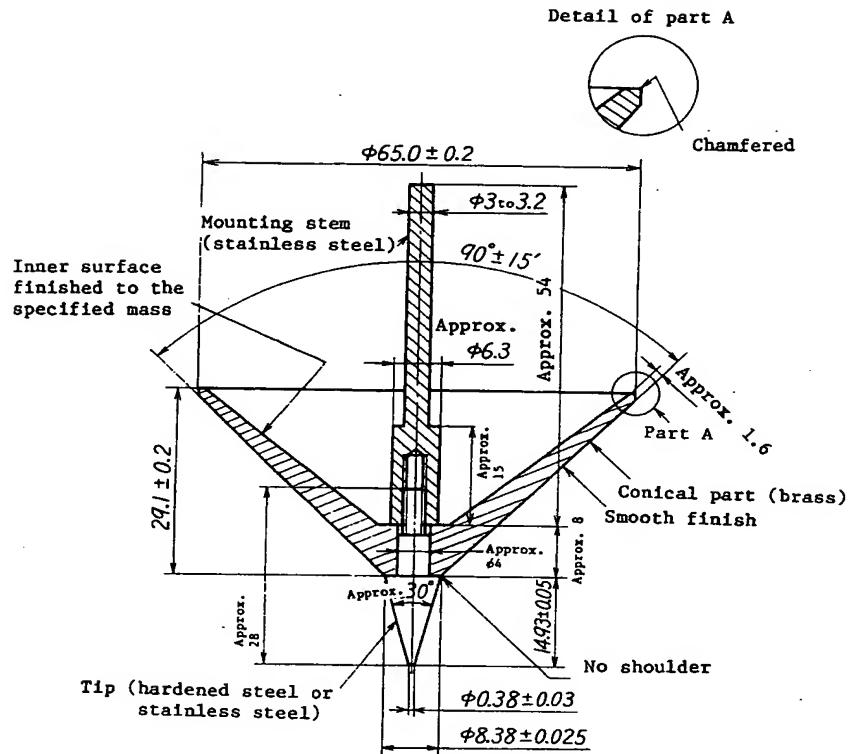


Fig. 3. Cone B

102.5  $\pm$  0.05 g in mass

Unit: mm

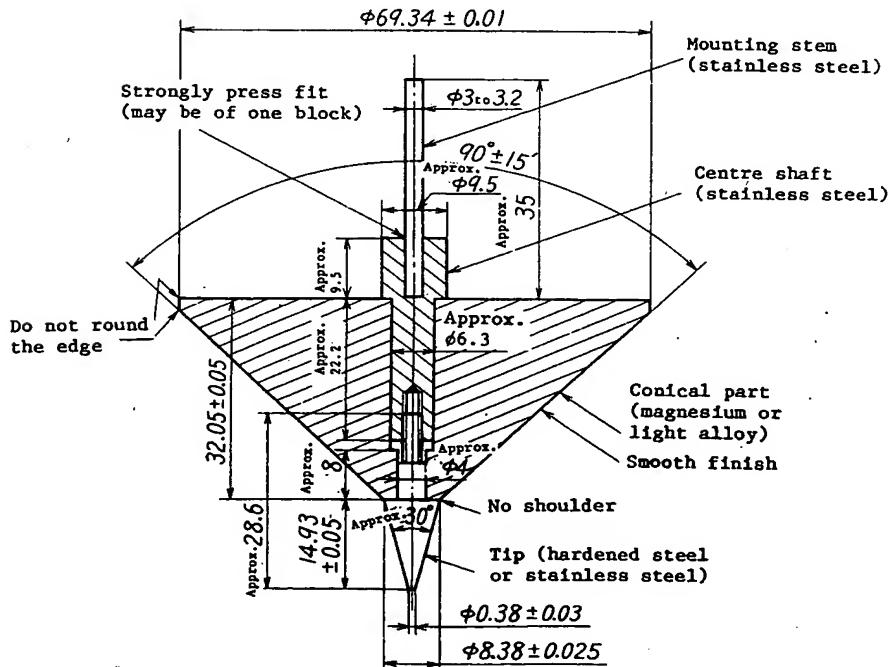


Fig. 4. Grease worker (An example)

Unit: mm

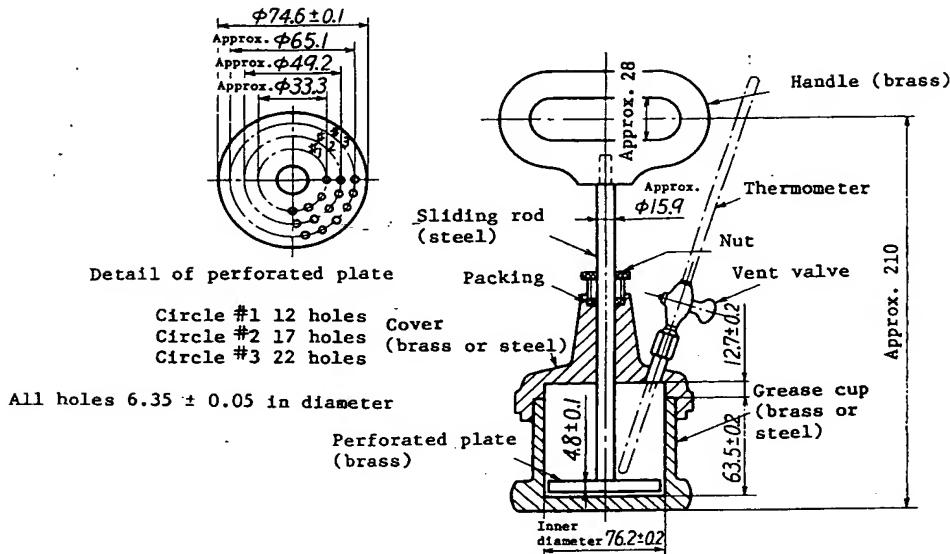
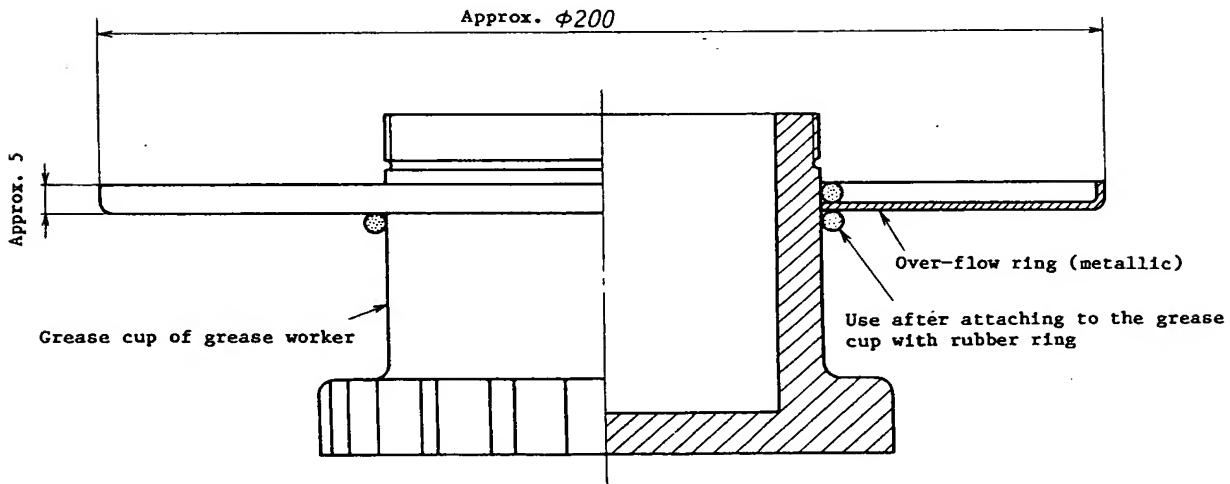


Fig. 5. Over-flow ring (An example)

Unit: mm



- (6) Motor driven grease working apparatus This apparatus shall have the construction as shown in Fig. 6, and shall give the stroke of 67 mm to 69 mm up and down at a rate of  $60 \pm 10$  double strokes per min to the perforated plate of grease worker by a motor.
- (7) Spatula This shall be made of stainless steel with a grip, shall have about 25 mm width and about 150 mm length. Its top end shall be of square shape.

(8) Cutter An example of the cutter is as shown in Fig. 7, and shall be equipped with a sharp blade of hardened steel or stainless steel.

(9) Thermostatic water bath The bath shall be capable of maintaining water temperature at  $25 \pm 0.5^{\circ}\text{C}$ , have a capacity of 30 l or more and suitable shelves for immersing the grease worker in the water bath.

Fig. 6. Motor driven grease working apparatus  
(Capable of mounting double workers) (An example)

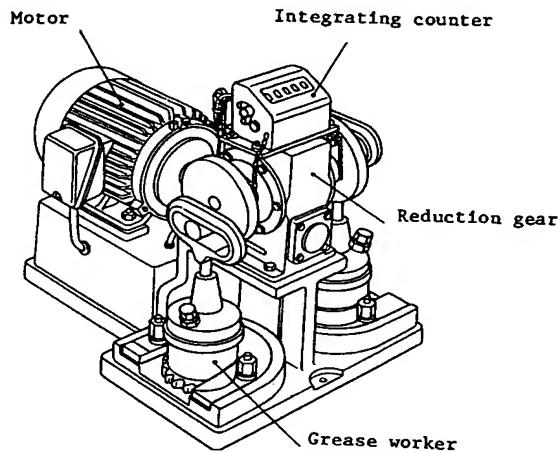
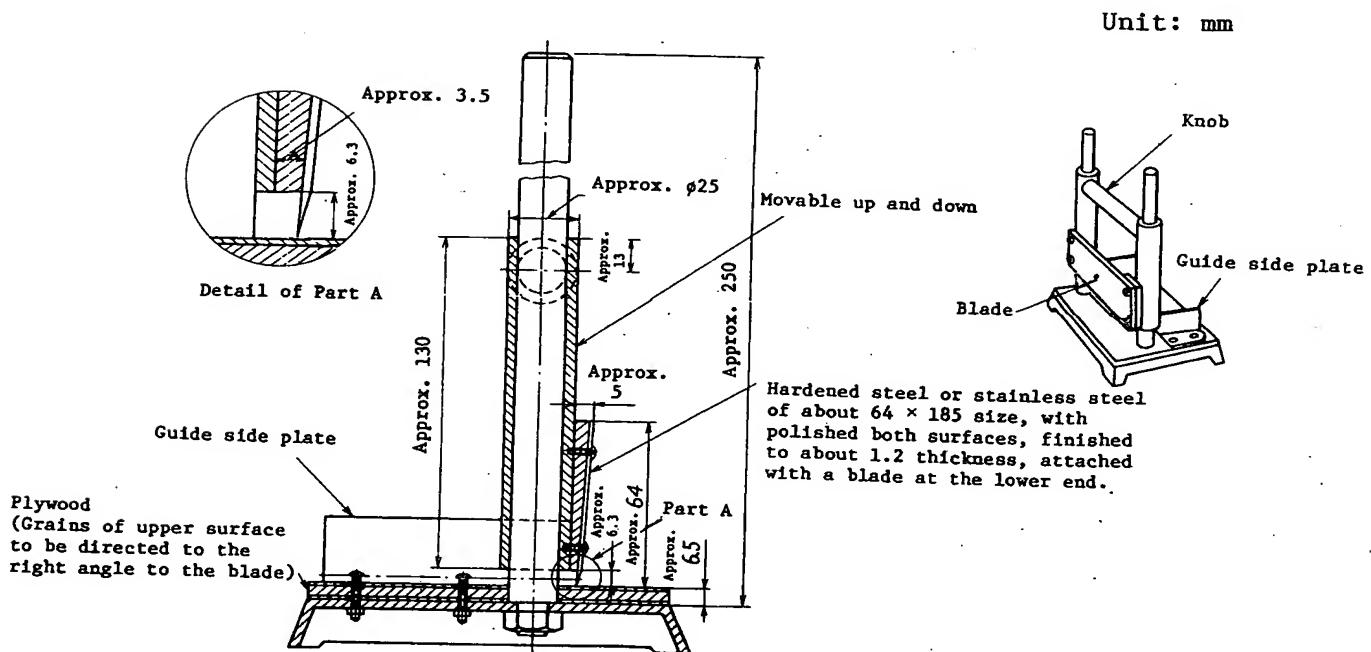


Fig. 7. Cutter (An example)



5.3.3 Measuring method for worked penetration The measuring method for worked penetration shall be as follows.

(1) Preparation of sample Preparation of the sample shall be made as follows:

(a) Prepare the sample about 500 g which will be enough to fill up the cup of the grease worker, fill the clean cup of the grease worker with the sample with a spatula with care not to allow air to enter into the sample, and mound it to mountain shape so that the central part will be about 10 mm higher than the rim of the cup. Then open the vent valve on the cover of the grease worker, assemble the grease worker, depress the perforated plate to the bottom of the cup, insert a thermometer through the vent valve to maintain the tip of the thermometer at the middle of the cup. Immerse the grease worker in the thermostatic water bath<sup>(4)</sup> kept at 25°C up to the lower rim of the lid of the cup comes beneath the water level<sup>(5)</sup>, and leave the cup still until the temperature of the sample reaches 25.0 ± 0.5°C<sup>(6)</sup>.

Notes (4) Instead of the thermostatic water bath, a thermostatic room or thermostatic air bath may be used, in such a case, leaving time shall be prolonged enough.

(5) When the grease worker is immersed in the thermostatic water bath, a grease worker which does not admit the entrance of water shall be used, or the entrance of water shall be prevented by putting the grease worker in polyethylene bag.

(6) It will require about 2 h for the sample to reach 25°C. In the case where the temperature of sample prior to immersing into the thermostatic water bath is 17°C or lower, or 33°C or higher, the leaving period of time in the thermostatic water bath shall be extended sufficiently.

(b) Then take out the grease worker from the thermostatic water bath, wipe off outer water, pull out the thermometer, close the vent valve, equipping the worker to motor driven grease working apparatus and work the sample<sup>(7)</sup> by moving the perforated plate up and down 60 double strokes for about 1 min, and return the perforated plate to the top. Then open the vent valve, dismount the cover and the perforated plate, remove the adhering sample as much as possible and return it to the cup. As the worked penetration of the sample may change on standing as it is, immediately proceed to the following procedures (c) and (2).

Note (7) Other working method may be used.

(c) Beat the cup of grease worker strongly to a suitable workbench to fill the air pockets generated by working for the purpose of getting homogeneity of the worked sample. Then take out a most part of the sample with a spatula to let the sample of the bottom part come out to the surface<sup>(8)</sup>.

Then for the removal of air bubbles, beat the cup strongly to the workbench. At this time, pack the over-flowed sample with the spatula. Scrape off the excess sample<sup>(9)</sup> extending above the rim of the cup and flatten the surface by moving the blade of the spatula, held inclined toward the direction of motion by an angle of about 45°, along the rim of the cup as shown in Fig. 8. From that time on, care shall be taken not to touch the surface of sample with the spatula or the like before measurement.

Fig. 8. Movement way of spatula



Notes (8) Care shall be taken not to work a sample beyond necessity.

(9) The sample having been removed from the cup shall be kept to fill the cup for the succeeding measurements.

(2) Measurement of penetration Measurement of penetration shall be done as follows.

- (a) Before the measurement, wipe the cone with a clean cloth with care not to bend the holder, and wipe off grease, oil and others adhering onto the holder, if any.
- (b) Put the cup containing the sample prepared in (1) on the sample table of penetrometer, ascertaining that the cup does not oscillate. After setting the position of the cone to zero point of dial gauge, let the tip of the cone approach to the surface of the sample by moving either the cone or the sample table up and down, let the tip of the cone contact the centre of the sample surface by operating the knob for fine adjustment with care.

Then press the retainer quickly to let the cone penetrate into the sample for  $5.0 \pm 0.1$  s<sup>(10)</sup>. Press down quietly till the rack for the measurement stops, and read the indication of the pointer in integer<sup>(11)</sup>.

Note (10) Prior to the measurement of penetration, start a stopwatch, instantly press the retainer at the same time when the second hand indicates arbitrary point, and release after elapse of specified measuring time.

Note <sup>(11)</sup> Especially in the case of measurement of soft sample, keep the outer surface of the cup clean and fit with over-flow ring so that the sample extruded by the cone may be returned to the cup for the succeeding measurement.

- (c) Return the sample which has been removed with the spatula and extruded sample by a cone to the cup of the grease worker.
- (d) Immediately repeat the procedures (1)(c) and (2)(a) to (c), and further measure the penetration two times.
- (3) Result For the same sample, round off the average of three measured results obtained in (2) to an integer in accordance with JIS Z 8401, and take it as the test result.

5.3.4 Measuring method for unworked penetration The measuring method for unworked penetration shall be as follows.

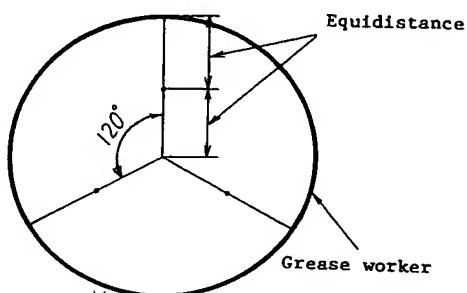
- (1) Preparation of sample Preparation of sample shall be made as follows:
  - (a) Prepare about 500 g for the sample having unworked penetration of 200 or under, and about 1500 g for the sample having unworked penetration exceeding 200, and those samples shall be contained in a suitable vessel with a lid.
  - (b) Immerse the sample in the container with a lid together with the assembled empty grease worker<sup>(12)</sup> in the thermostatic water bath kept at 25°C, and leave them still for the time enough for the sample to reach a temperature of  $25.0 \pm 0.5$  °C.
- (c) Note <sup>(12)</sup> A container having the same inner diameter as that of the cup of the grease worker may be used.
- (d) Take out the container with a lid and the grease worker from thermostatic water bath, and wipe off water adhering onto their outer surfaces.
- (e) Transfer the sample from the container with lid into the cup of the grease worker preferably in one lump. During the time, remove the air involved in the sample by beating the cup of the grease worker onto a suitable workbench, and fill up the sample in the cup with care not to disturb the sample.
- (f) Giving the surface of a spatula about 45 degrees inclination to the direction of moving as shown in Fig. 8, move the spatula along the upper rim of the cup, remove the excess sample, and flatten the surface of sample. Hereafter, prior to the measurement, the surface of sample shall not be contacted by a spatula or the like.

- (2) Measurement of penetration Measurement of penetration shall be made as follows:

- (a) For the sample whose penetration exceeds 200, measure the penetration at the centre of sample surface in accordance with the procedures specified in 5.3.3 (2)(a) and (b).

- (b) Take out the sample having been finished the measurement from the cup, wipe the cup with a clean cloth to make it clean, then put the sample prepared in (1)(b) into the cup according to the procedures in (1)(d) and (e). Thereafter, measure the penetration further two times according to (a).
- (c) For the sample whose penetration is 200 or under, measure the penetration at three different positions respectively in the same cup as shown in Fig. 9, in accordance with the procedures specified in 5.3.3 (2)(a) and (b).

Fig. 9. Measuring position of penetration (- mark)



- (3) Result For the same sample, round off the average value of three measured results obtained in (2) to an integer in accordance with JIS Z 8401, and take it as the test result.

#### 5.3.5 Measuring method for prolonged worked penetration The measuring method for prolonged worked penetration shall be as follows.

##### (1) Preparation of sample Preparation of sample shall be made as follows:

- (a) Prepare a sample of about 500 g which is enough to fill up the cup of a grease worker, and leave it still until the sample reaches the same temperature as that of testing place.

Remarks: The room temperature of testing place should preferably be kept in the range of 15°C to 30°C.

- (b) Fill the cup of clean grease worker with the sample with a spatula with care not to make air pockets in the sample and mound it to a mountain shape so that the central part will be about 10 mm or higher than the rim of the cup, then open the vent valve on the cover of the grease worker, and assemble the grease worker. Then depress the perforated plate to the bottom of the cup, close the vent valve, mount the grease worker specified in 5.11.2 (1) to a motor driven working apparatus, and work the sample to the specified number of turns.

Remarks: The specified number of turns shall be hundred thousand turns, which shall be subject to agreement between the purchaser and the manufacturer.

(2) Measurement of penetration Dismount the grease worker from the motor driven working apparatus, depress the perforated plate to the bottom of the cup, open the vent valve on the cover of the grease worker, and insert a thermometer through the valve to hold the tip at the centre of the cup. Immerse<sup>(5)</sup> the grease worker in the thermostatic water bath<sup>(4)</sup> kept at 25°C to make the lower rim of the lid be beneath the water level, and leave it still until the sample reaches a temperature of 25.0 ± 0.5°C<sup>(6)</sup>. Hereafter, measure the worked penetration by the method specified in 5.3.3 (1)(b), (c), and 5.3.3 (2).

(3) Result For the same sample, round off the average value of three measured results obtained in (2) to an integer in accordance with JIS Z 8401, and take it as the test result.

5.3.6 Measuring method for undisturbed penetration The measuring method for undisturbed penetration shall be as follows.

(1) Preparation of sample Preparation of the sample shall be made as follows:

- Prepare about 500 g for the sample having unworked penetration of 200 or under, and about 1500 g for the sample having unworked penetration exceeding 200.
- The sample shall be filled fully into the cup of worker<sup>(12)</sup> with care not to make air bubbles in the sample, and then flatten the surface of sample in accordance with 5.3.4 (1)(e).
- When the penetration is 200 or under, one sample shall be prepared, and for grease of which penetration exceeds 200, three samples shall be prepared.
- Put on a suitable cover with care not to touch the surface of sample, and store or stand as it is for the specified hours<sup>(13)</sup>.

Note (13) As to the hours and the place of storing or standing, the agreement shall be made between the purchaser and supplier.

- Sink the cup containing the sample in a thermostatic water bath kept at 25°C to such depth that the rim of the cup is about 25 mm above the water surface<sup>(4)</sup>. Leave for 2 h with a cover over the water bath and also with care not to allow water to enter the sample. Then take the cup out of the thermostatic water bath, and wipe off the water adhering onto its outer surface.

(2) Measurement of penetration Measurement of penetration shall be made as follows.

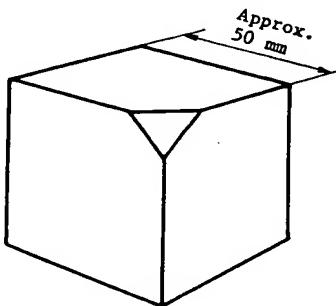
- When the penetration of the sample exceeds 200, measurement of penetration shall be made on three samples prepared in accordance with the procedures specified in 5.3.3 (2)(a) and (b), respectively.
- When the penetration is 200 or under, the measurement of penetration shall be made in accordance with 5.3.4 (2)(c).

(3) Result For the same sample, round off the average value of three test results obtained in (2) to an integer in accordance with JIS Z 8401, and take it as the test result.

5.3.7 Measuring method for block penetration The measuring method for block penetration shall be as follows:

(1) Preparation of sample Prepare the sample bigger than 50 mm cube, which is hard enough to maintain a shape, cut the sample into about 50 mm cube with the specified cutter at room temperature, and further slice three planes which have an arbitrary common apex by about 1.5 mm in thickness each to form the measuring surface. Cut the apex a little for the mark, as shown in Fig. 10. Put the sample thus prepared into the vessel tightly sealed, with care not to touch the measuring surface by fingers, leave it in the thermostatic water bath<sup>(4)</sup> kept at 25°C for an hour or more to make the sample temperature up to  $25.0 \pm 0.5^{\circ}\text{C}$ .

Fig. 10. Sample for measurement of block penetration



(2) Measurement of penetration Measurement of penetration shall be made as follows.

(a) Before the measurement, wipe the cone with a clean cloth with care not to bend the holder, and wipe off grease, oil and others adhering onto the holder.

(b) Place the sample on the sample table of penetrometer with one measuring surface upward, push the sample lightly to make it horizontal, and ascertain it does not move during measurement. After setting the position of cone to zero point of dial gauge, let the tip of cone approach to the surface of the sample by moving either the cone or sample table up and down, and with operating carefully the knob for fine adjustment, let the tip of cone contact the measuring surface at a point 10 mm or more apart from the edge.

Then, push the retainer quickly, penetrate the cone into the sample for  $5.0 \pm 0.1 \text{ s}^{(10)}$ , and then push down till the rack of measurement stops and read the indication of pointer in integer.

(c) Measure the penetration three times on the same measuring surface at the points 10 mm or more away from each other according to the method specified in (a) to (b). When an air hole or crack is found, measurement shall be made further at a 10 mm or more distant points.

(d) When the difference in penetration on the same surface exceeds 3, repeat the measurement till three values of measurement within 3 are obtained.

This average value shall be regarded as the result obtained on the same sample surface, and the same measurements shall be made on other two surfaces.

(3) Result Round off the average value of measured results on three surfaces to an integer in accordance with JIS Z 8401, and take it as the test result.

5.3.8 Precision The precision shall be as follows:

(1) Repeatability The tolerance on the difference between two successive test results, obtained by the same person with the same apparatus under the same test conditions on identical test samples shall be given in Table 16.

Table 16. Repeatability

Penetration	Range of penetration	Tolerance
Worked penetration	130 to 475	5
Unworked penetration	85 to 475	6
Prolonged worked penetration	Unspecified	Unspecified
Undisturbed penetration	Unspecified	Unspecified
Block penetration	85 max.	3

(2) Reproducibility The tolerance on the difference between two single and independent test results, obtained by different persons with different apparatuses in different two laboratories on identical test sample shall be given in Table 17.

Table 17. Reproducibility

Penetration	Range of penetration	Tolerance
Worked penetration	130 to 475	14
Unworked penetration	85 to 475	18
Prolonged worked penetration	Unspecified	Unspecified
Undisturbed penetration	Unspecified	Unspecified
Block penetration	85 max.	7

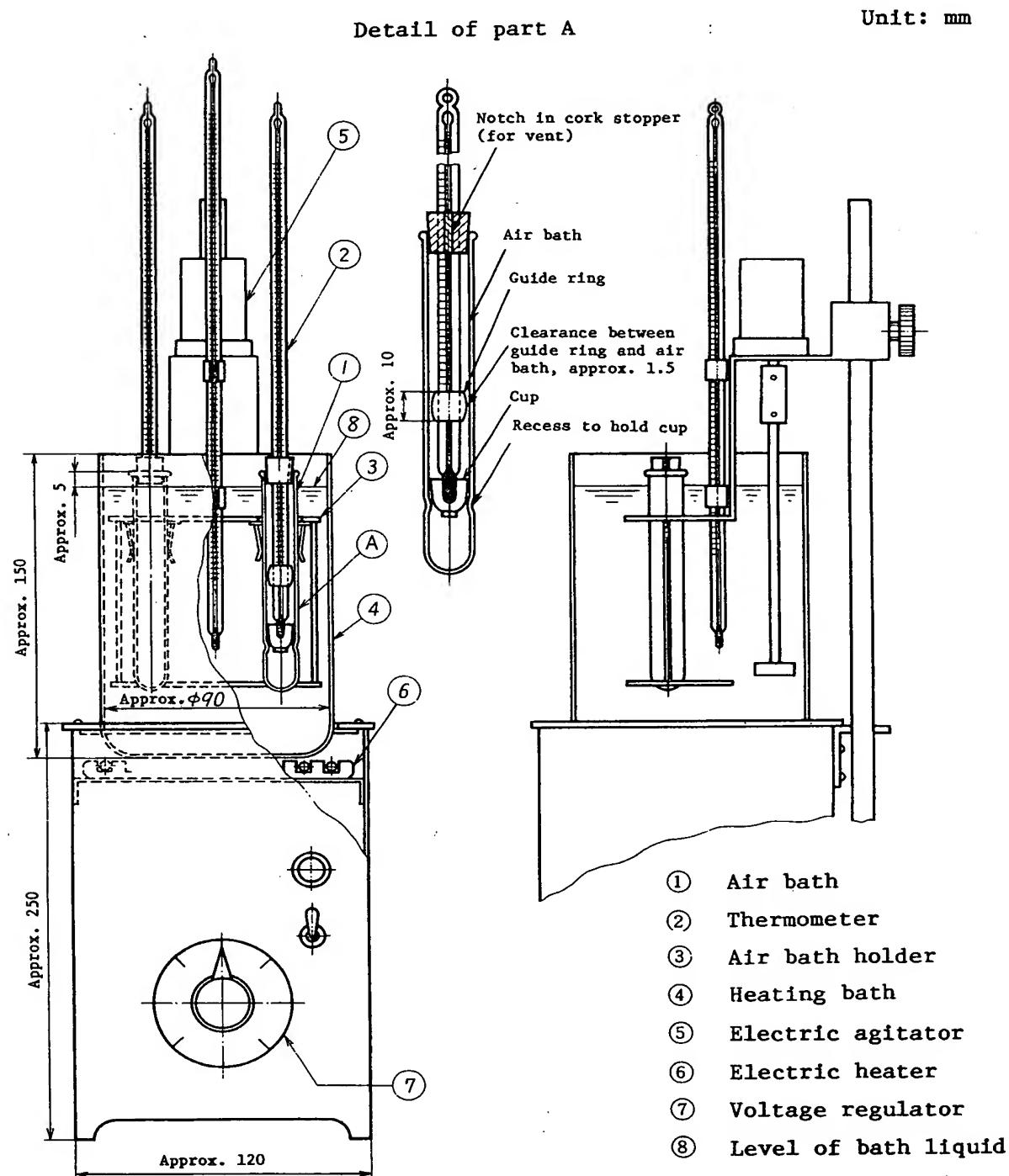
Remarks: When the test result exceeds the tolerance, the result shall be treated in accordance with JIS Z 8402.

#### 5.4 Test method for dropping point

5.4.1 Summary of the test method Fill a cup with sample, put the cup in an air bath. Insert a thermometer, heat under the specified conditions in a heating bath, and obtain the dropping point from the indication of thermometer at the time when the sample begins to drop from the rim of the cup.

5.4.2 Testing apparatus for dropping point The testing apparatus for dropping point shall be composed of the following (1) to (5). Example of the construction of the testing apparatus for dropping point shall be given in Fig. 11.

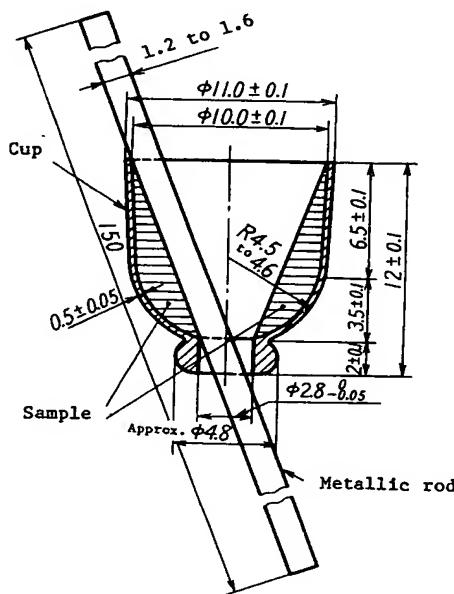
Fig. 11. An example of construction of testing apparatus for dropping point (for double mounting)



(1) Cup and metallic rod The cup and metallic rod shall be as shown in Fig. 12 in shape and dimensions. Material of the cup shall be brass, plated with chromium. Metallic rod shall be of steel, polished and straight.

Fig. 12. Cup and metallic rod

Unit: mm



(2) Air bath The air bath shall be as specified in Fig. 173 of JIS K 2839.

(3) Heating bath The heating bath shall be as specified in Fig. 174 of JIS K 2839, and it shall be equipped with the air bath holder and a suitable agitator as shown in Fig. 11.

Remarks: For the liquid of bath, such as heat resistant silicone oil which has good thermal stability with thin colourant shall preferably be used.

(4) Heater Heater shall be a gas burner or an electric heater capable of raising the temperature of bath liquid at an arbitrary rate ranging from 1°C to 7.5°C per minute.

(5) Thermometer Thermometer shall be of number DP 38 (for air bath/for heating bath) specified in JIS B 7410, and shall be equipped with a cork stopper with a notch for vent and a guide ring as shown in Detail of Part A in Fig. 11.

5.4.3 Preparation for the test Preparation for the test shall be made as follows:

- (1) Put a cork stopper and guide ring onto the thermometer for air bath, assemble the apparatus as shown in Fig. 11, and make the lower end of mercury bulb of thermometer positioned about 3 mm above the inner bottom of the cup.
- (2) Remove the cork stopper and the thermometer fitted in (1) from the air bath with care not to shift the position, then take out the cup from the air bath.
- (3) Fill the cup with the sample from the bigger mouth with care not to work the sample as possible. Remove the sample extruded from both ends with a spatula. Insert the metallic rod from the smaller mouth to protrude about 25 mm, push the rod to the cup to allow the rod to contact the rims of upper and lower edges. Turning the cup spirally while pressing the rod, remove the sample in conical form from the cup. The sample remaining in the cup shall be of form as shown in Fig. 12, and its surface shall be smooth.

5.4.4 Test procedure Test procedure shall be as follows:

- (1) Put the cup in the air bath, keep horizontal at the recess, and insert the thermometer for the air bath fitted in 5.4.3 (1) with care not to allow the mercury bulb to contact the sample. Put the air bath in heating bath.

Adjust the liquid amount preliminary so that the liquid level attains within 5 mm from the top end of the air bath when the temperature reaches the dropping point of sample.

Suspend the thermometer for heating bath in the heating bath so as the lower end of its bulb to be at about equal height as that of thermometer for air bath.

- (2) Start heating with agitating the heating bath, raise the temperature at a rate of 5°C to 7.5°C per min until the heating bath reaches the temperature 20°C lower than the expected dropping point of the sample. When reaching the said temperature, weaken the heating, and adjust heating so that during further 3.0°C elevation of bath temperature, the temperature of air bath approaches heating bath temperature within 2.5°C. Then raise the temperature of heating bath at a rate of 1°C to 2°C per min so that the difference of the temperature between the air bath and the heating bath falls within 1°C to 2°C. Record in integer the indications of both thermometers at the time when the sample drops from the rim of the cup or arrives at the bottom of air bath.

Remarks: For the greases whose dropping points are nearly the same, plural samples may be tested in the same heating bath.

5.4.5 Result Round off the average value of indications of both thermometers recorded in 5.4.4 (2) to an integer in accordance with JIS Z 8401, and take it as the test result.

#### 5.4.6 Precision

(1) Repeatability When the duplicate tests are made for the same sample by the same person with the same testing apparatus in the same laboratory at different times or on different days, the difference on test results shall not exceed the following tolerance.

Tolerance 7°C

(2) Reproducibility The difference on test results between two single and independent tests made for the same sample by different persons, with different testing apparatus in two different laboratories shall not exceed the following tolerance.

Tolerance 13°C

Remarks 1. When the test result exceeds the tolerance, the result shall be treated in accordance with JIS Z 8402.

2. Samples containing aluminium soap are apt to exceed the tolerance abovementioned.

#### 5.5 Test method for copper corrosion

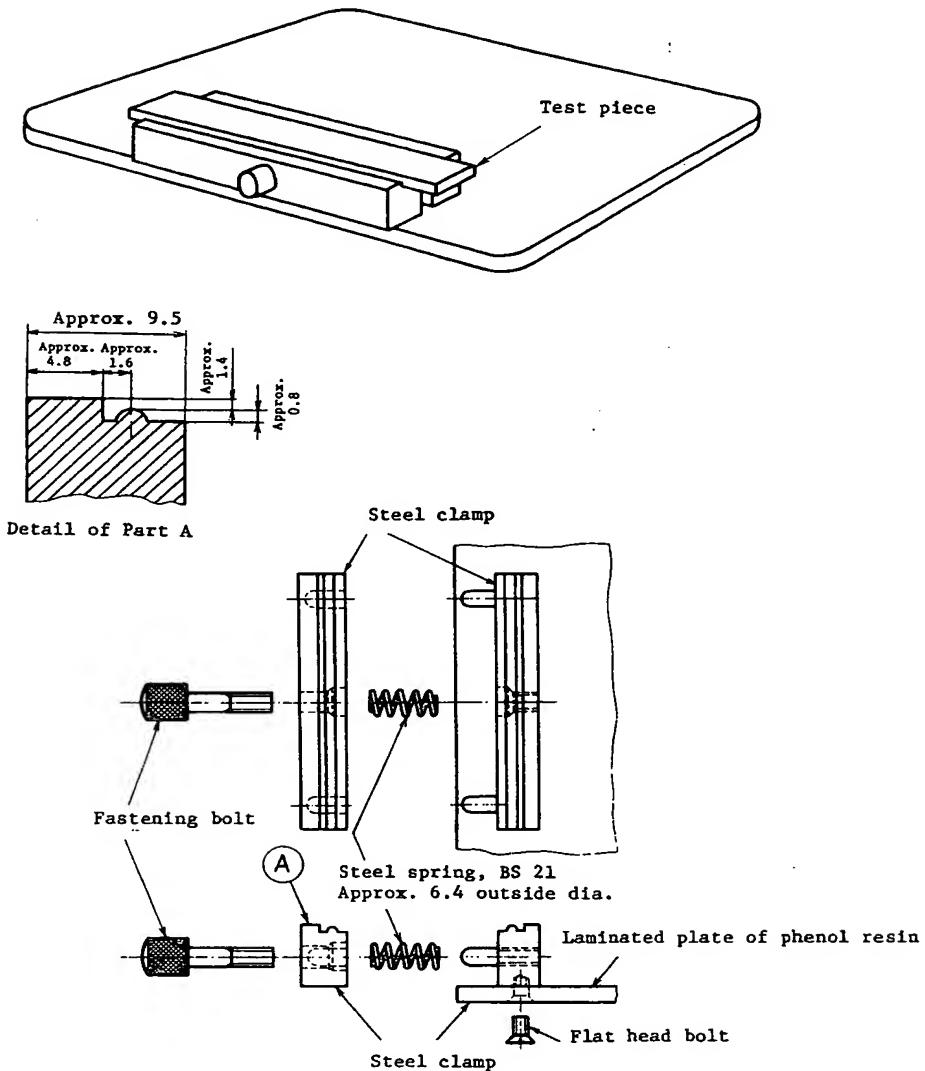
5.5.1 Summary of the test method Immerse a polished copper plate in the grease, and examine the presence of discoloration on the copper plate after keeping for 24 h at room temperature (in the case of Method A) or at 100°C (in the case of Method B).

5.5.2 Apparatus and devices The apparatus and devices shall be composed of the following (1) to (7).

- (1) Test tube A glass test tube specified in Fig. 111 of JIS K 2839, or a beaker.
- (2) Thermostatic air bath An electric heating thermostatic air bath capable of keeping temperature at  $100 \pm 1.0^\circ\text{C}$ , equipped with a suitable holder which is able to hold test tube vertically.
- (3) Holder for polishing A holder of shape and dimensions shown in Fig. 13, to be used to fix the test piece when polishing. (Refer to JIS K 2513.)

Fig. 13. Holder for polishing (An example)

Unit: mm



- (4) Thermometer No. 42 thermometer (thermometer for specific gravity floating method) specified in JIS B 7410.
- (5) Test piece Made of C1100P, C1201P or C1220P specified in JIS H 3100, having about 75 mm in length, about 12.5 mm in width and 1.5 mm to 3.0 mm in thickness.

Repeated use of a test piece is allowed, provided that the piece has not any unremovable deep flaw nor deformed surface.

- (6) Abrasives The abrasives specified as follows shall be used.

- (a) Abrasive paper of silicon carbide or fused alumina specified in JIS R 6252.

- (b) Abrasive cloth of fused alumina or silicon carbide specified in JIS R 6251.
- (c) Artificial abrasive (C or GC) of grain size number 150 specified in JIS R 6111.
- (d) Pharmacopoeia absorbent cotton

(7) Solvent for cleaning Guaranteed grade acetone specified in JIS K 8034.

5.5.3 Preparation of test Preparation of test shall be made as follows:

(1) Preliminary polishing of test piece Remove the flaws on whole surface of test piece by means of abrasive paper or abrasive cloth of grain size specified in 5.5.2 (6)<sup>(14)</sup>. Then polish with No. 240 grain size abrasive paper or cloth, and immediately after cleaning the test piece by immersing in solvent, proceed to finish polishing. When the finish polishing is unable to be made in succession, the test piece shall be immersed still in the solvent.

Note <sup>(14)</sup> Place abrasive paper or cloth on a flat plate, wet it with the solvent, put the test piece on it, press the test piece with an ashless filter paper, and polish the test piece by circular movement.

(2) Finish polishing of test piece Take the test piece out of the solvent, put it between ashless filter papers and hold with hand, first polish both end surfaces with absorbent cotton slightly wetted with solvent and putting artificial abrasive on it, then polish the both side surfaces. Further rub strongly with fresh absorbent cotton alone. Hereafter, handle the test piece with a stainless steel pincette with care not to allow finger to touch the piece directly. Fix the test piece to the holder for polishing, polish the both principal surfaces of test piece in its longitudinal direction by the use of absorbent cotton putting artificial abrasive on it. The polishing shall be made uniformly from one end to another of the test piece with care not to round the edges. Finally rub strongly with absorbent cotton alone until no stain is found on fresh absorbent cotton. Within one min put the test piece in the sample.

(3) Cleaning of test tube Wash to clean the test tube by immersing in a specified cleaning solution in 5.5.2 (7), wash with running water until the cleaning solution is gone out, then rinse with distilled water, and dry.

5.5.4 Test procedures Test procedures shall be as follows:

(1) Place a sample in a test tube to about 90 mm depth and bury the test piece until the upper edge sinks in the sample with care not to involve air bubbles<sup>(15)</sup>.

Note <sup>(15)</sup> It should be preferable to coat previously the same sample on the whole surface of the test piece.

(2) In the case of Method A, leave the test tube at room temperature for 24 h.

In the case of Method B, hold the test tube vertically, and place it in thermostatic air bath kept at  $100 \pm 1^\circ\text{C}$  for 24 h. Take it out and leave it for cooling to room temperature.

(3) Pull out the copper plate from the test tube with a stainless steel pincette, and clean it with solvent.

5.5.5 Result Examine the presence of change in colour to green or black on the copper plate.

#### 5.6 Test method for evaporation loss

5.6.1 Summary of test method Apply heated air on the surface of a sample for 22 h in a bath kept at a specified temperature. Calculate the evaporation loss from the loss in mass of the sample.

5.6.2 Apparatus and device The apparatus and device shall be composed of the following items (1) to (6).

(1) Evaporator An example of the evaporator is given in Fig. 14. Its outer cylinder, lid, exhaust tube and outlet shall be made of stainless steel (SUS304), and the air preheating tube shall be made of tin plated copper tube.

(2) Sample container and cover An example of the sample container and cover is shown in Fig. 15. The material of those shall be of stainless steel and the mass of the sample container shall be 200 g or under. For easy detaching of the sample container in charging and weighing the sample, all of the container, cover, and exhaust tube shall be of threaded type.

(3) Air supplying apparatus The air supplying apparatus shall be capable of supplying air at the specified flow rate to the evaporator through a filter device. The filter device shall be a tube of about 25 mm diameter and about 400 mm length which is stuffed with glass wool.

(4) Thermostatic bath The thermostatic bath shall be deep enough to be able to immerse the evaporator to a suitable depth, and shall be capable of maintaining the temperature within  $\pm 0.5^\circ\text{C}$  of specified temperature, and shall be equipped with such a regulator as to adjust the bath temperature within  $0.5^\circ\text{C}$ .

Remarks: The liquid of thermostatic bath shall have a sufficient thermal stability as silicon oil.

(5) Thermometer The following thermometers specified in JIS B 7410 shall be used.

For the test at  $99^\circ\text{C}$ , No. 6 thermometer

For the test over  $99^\circ\text{C}$ , No. 26 thermometer

(6) Flow meter This shall be capable of measuring the passing amount of  $2.58 \pm 0.02$  g/min (2 l/min under standard conditions) at  $15^\circ\text{C}$  to  $29^\circ\text{C}$  (such as rotor meter) equipped with air regulating valve.

Fig. 14. Construction of evaporator (An example)

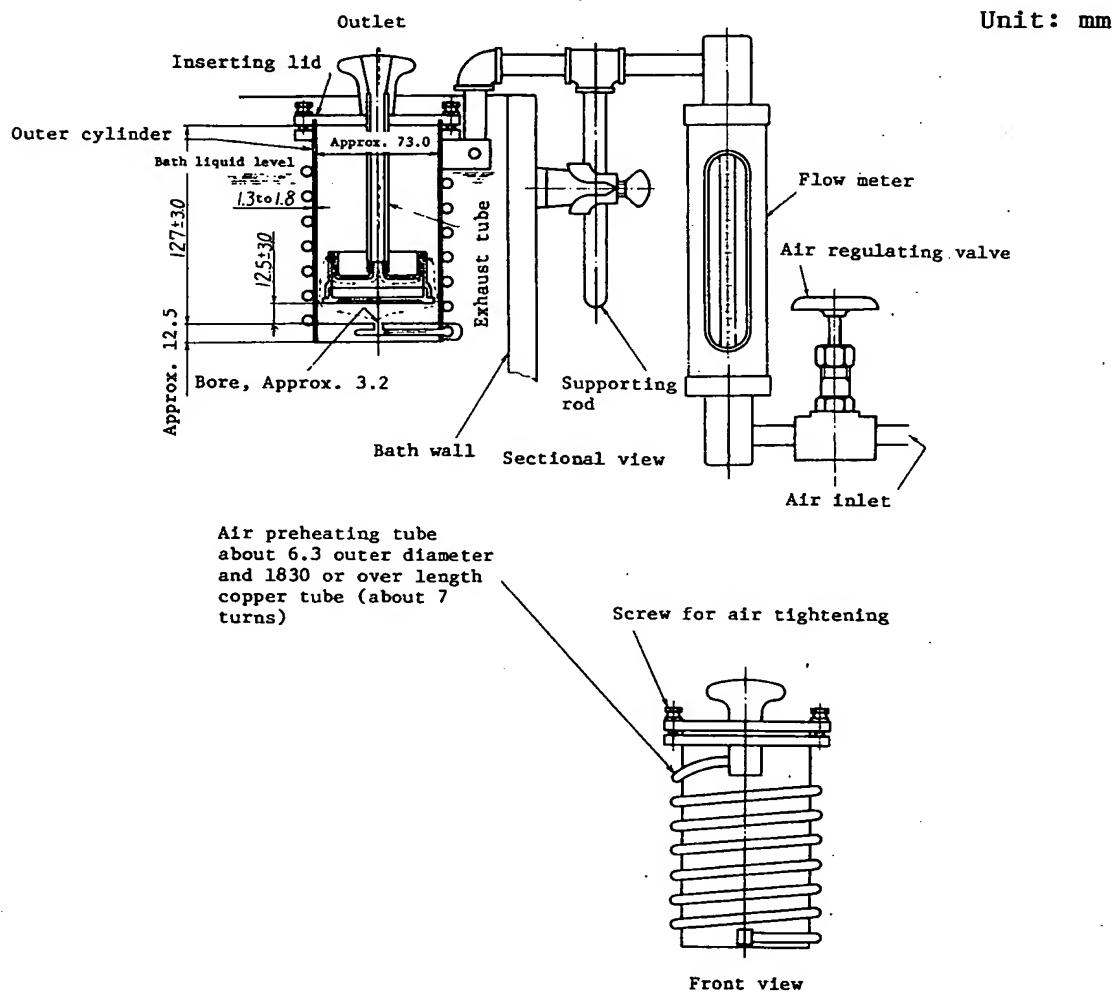
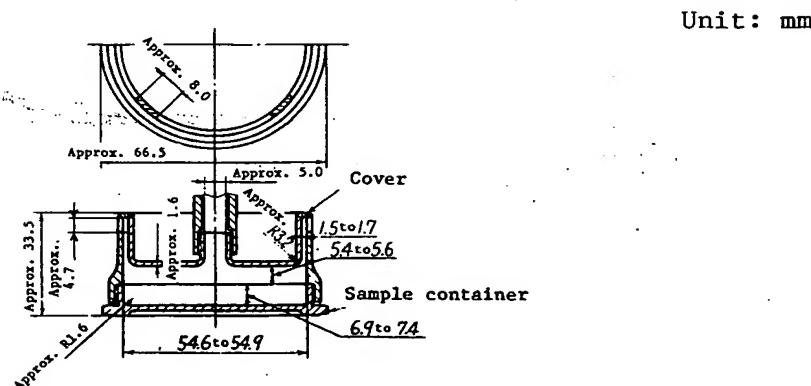


Fig. 15. Sample container and cover (An example)



**5.6.3 Test procedures** Test procedures shall be as follows:

- (1) Weigh a clean sample container together with the cover to the nearest 1 mg, and fill the sample with care not to allow air to enter. Flatten the surface along the rim of the container with a straight blade of a spatula. Remove the sample adhering onto the rim and the screw with a clean cloth. Screw the cover onto the container with care not to give flaws to the flattened surface. Obtain the mass of the sample to the nearest 1 mg.
- (2) Regulate the thermostatic bath containing the evaporator at test temperature  $\pm 0.5^{\circ}\text{C}$ , and leave it still at least for 30 min while sending clean air to the evaporator at a rate of  $2.58 \pm 0.02 \text{ g/min}$  (2 l/min under standard conditions). Then detach the lid, attach the tared sample container and the tared cover to the exhaust tube, and put the lid again, clamp strongly several fastening screws so as not to allow air to leak under the lid. Send air into the evaporator for 22 h  $\pm 5$  min.
- (3) After 22 h have elapsed, take out the container as it is attached with the cover, and leave it still in a desiccator for cooling to the room temperature. Weigh and obtain the mass of the sample to the nearest 1 mg.

**5.6.4 Calculation and result** Calculate the evaporation loss percentage according to the following formula, and express it in an average obtained by rounding off to two places of decimals from two test results obtained by 5.6.3 for the same sample.

$$W_L = \frac{W_s - W}{W_s} \times 100$$

where,  $W_L$  : evaporation loss percentage (mass %)

$W_s$  : mass of sample before test (g)

$W$  : mass of sample after test (g)

**5.6.5 Precision** The precision shall be as follows.

- (1) Repeatability The tolerance on the difference between two successive test results, obtained by the same person with the same apparatus in the same laboratory on identical test sample shall be as follows:

Tolerance: 2.5 % of the average

- (2) Reproducibility The tolerance on the difference between two single and independent test results, obtained by different persons with different apparatuses in different two laboratories on identical test sample shall be as follows:

Tolerance: 10 % of the average

Remarks: When the test result exceeds the tolerance, the result shall be treated in accordance with JIS Z 8402.

### 5.7 Test method for oil separation

5.7.1 Summary of test method Calculate the oil separation percentage from the mass of oil having separated from the sample in the conical wire gauze filter kept at the specified temperature in the specified time.

5.7.2 Apparatus and device The apparatus and device shall be composed of the following items (1) to (5).

(1) Conical wire gauze filter (hereafter referred to as "filter") The filter shall be of the shape and dimensions as shown in Figs 16 and 17, and the conical part shall be made of nickel wire gauze of 250  $\mu\text{m}$  in nominal size specified in JIS Z 8801. Nickel wire of about 0.8 mm diameter shall be brazed to the circumference of the upper rim, and a hanger of nickel wire of the same diameter shall be attached to it.

Fig. 16. Conical wire gauze filter

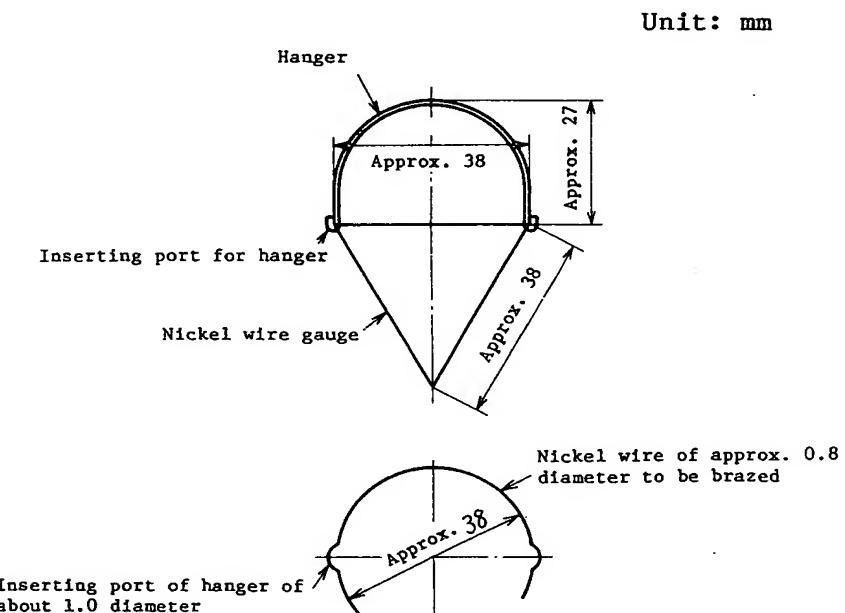
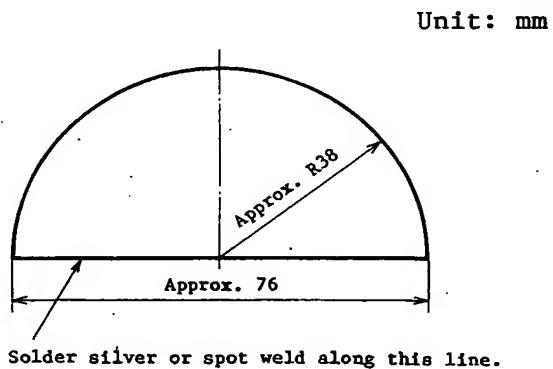


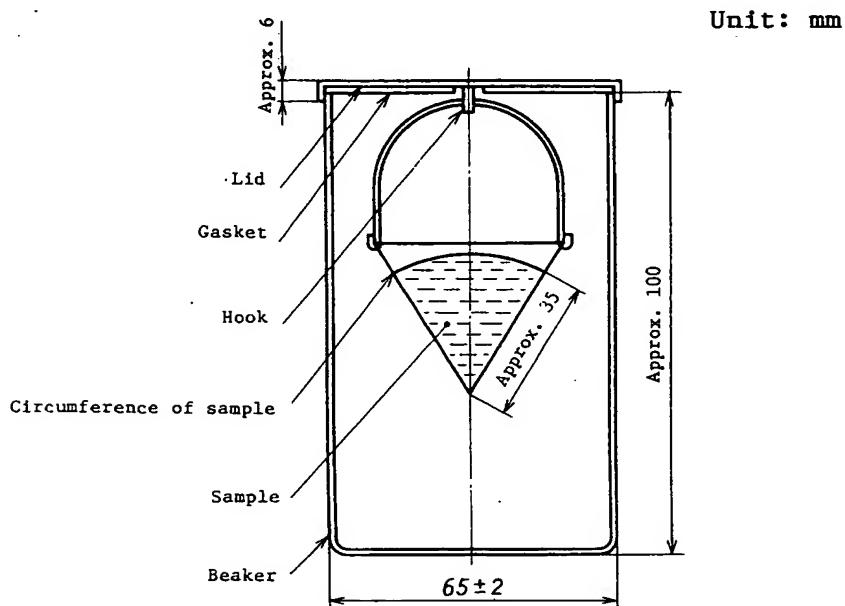
Fig. 17. Development drawing of conical wire gauze



(2) Beaker The beaker as specified in Fig. 177 of JIS K 2839.

(3) Lid A copper or brass made plate, about 1 mm thick, of the shape shown in Fig. 18. A copper or brass made hook of about 1.5 mm diameter shall be brazed near the centre of the inside surface.

Fig. 18. Assembly of oil separation tester (An example)



(4) Gasket The gasket shall have the same diameter as inner diameter of the lid, and shall be made of synthetic rubber of about 1.5 mm thickness with a hole of about 20 mm punched at its centre.

(5) Thermostatic air bath An electric heating type capable of keeping the specified temperature  $\pm 0.5^{\circ}\text{C}$ .

#### 5.7.3 Test procedures The procedures shall be as follows:

- (1) Depress the sample uniformly to the inside of the wire gauze with a spatula from the top end of the clean filter of known mass to the position of about 35 mm length so as to allow the sample to be extruded out of the wire gauze, then stuff the sample slowly with care not to allow air bubbles to enter. Then raise up the sample surface so as not to accumulate separated oil on the surface, and smoothen it with a spatula. Remove the sample extruded from the net of the filter by finger, and after adjusting the whole mass of the sample to be about 10 g, measure the mass to the nearest 0.01 g.
- (2) Suspend the filter containing the sample with the hook of the lid, put in a clean beaker of known mass, and place in the thermostatic air bath kept at the specified temperature  $\pm 0.5^{\circ}\text{C}$  for the specified hours. Then take out the beaker from the thermostatic air bath, leave it still in a desiccator to cool down to room temperature. By hitting the end of the filter slightly on the inside edge of the beaker, transfer the oil adhering on the tip of the cone to the beaker. Measure the mass of the separated oil to the nearest 0.01 g.

5.7.4 Calculation and result Calculate the oil separation percentage according to the following formula, round off the average of two measured results for the same sample obtained according to 5.7.3, to one place of decimal in accordance with JIS Z 8401 and take it as the test result.

$$A = \frac{C}{B} \times 100$$

where,  $A$  : oil separation percentage (mass %)

$B$  : mass of sample (g)

$C$  : mass of separated oil (g)

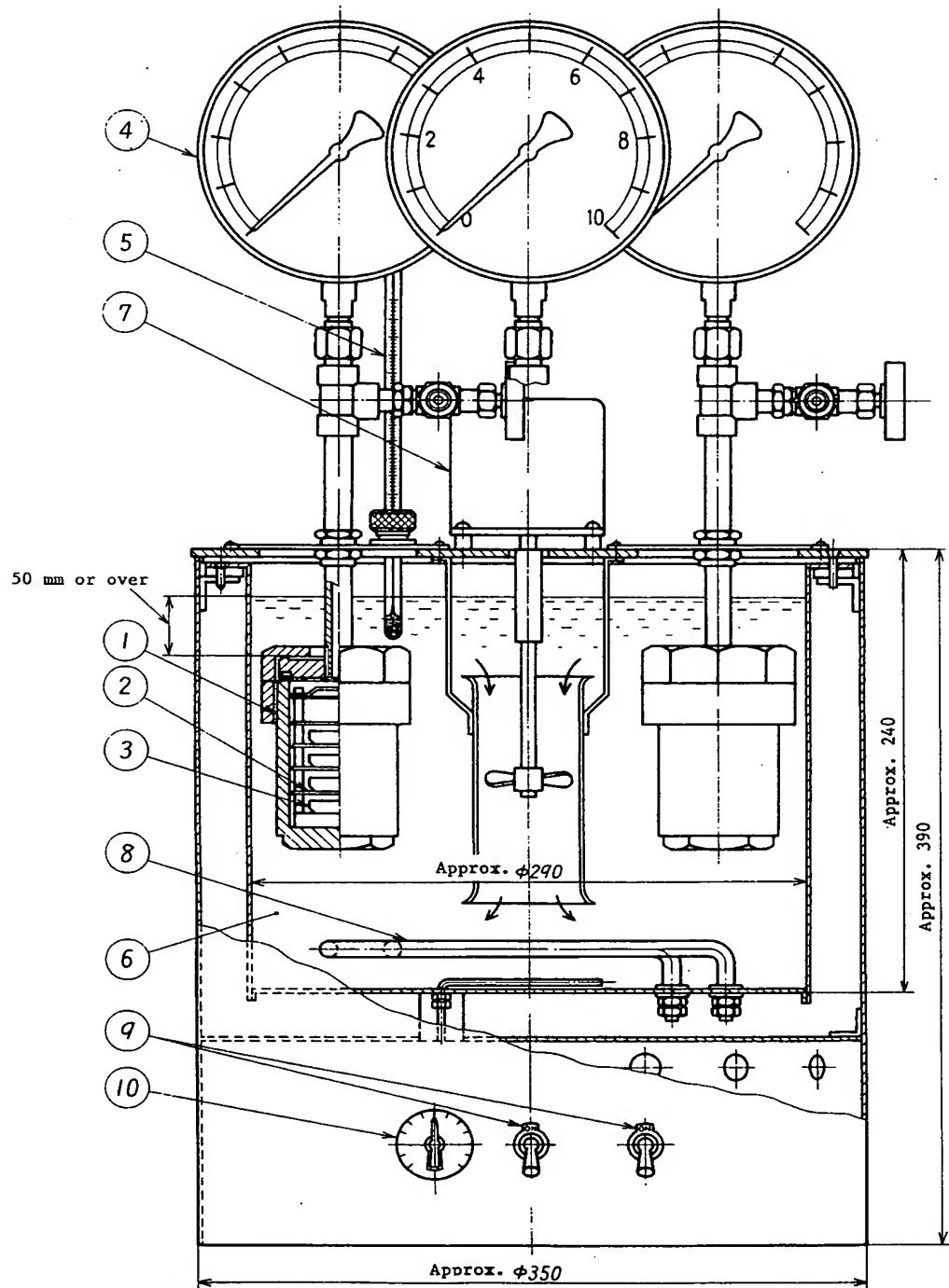
## 5.8 Test method for oxidation stability

5.8.1 Summary of test method Heat the sample at 99°C in a bomb under oxygen pressure of 0.76 MPa {7.7 kgf/cm<sup>2</sup>}, record the drop of oxygen pressure at every interval of definite time, and measure the oxygen pressure decrease at 100 h elapse.

5.8.2 Oxidation stability tester The oxidation stability tester shall be composed of the following items (1) to (8). An example of the oxygen stability tester is given in Fig. 19.

Fig. 19. Construction of oxidation stability tester (An example)

Unit: mm



① Bomb	⑥ Thermostatic oil bath
② Holder for sample container	⑦ Electric agitator
③ Sample container	⑧ Electric heater
④ Pressure gauge	⑨ Switch
⑤ Thermometer	⑩ Temperature regulator

(1) Bomb An example of the bomb is given in Fig. 20. The bomb shall be made of anti-corrosive metal with pressure-proof and airtightness, and be composed of the bomb body, needle valve, the lid with pipe for attaching pressure gauge, and nuts and gaskets for fastening the bomb. The materials of those shall be as specified in Table 18 or equivalent or superior to them, shall withstand the hydraulic test of 3.92 MPa{40 kgf/cm<sup>2</sup>}, and a pressure drop shall not be found when placed still in thermostatic bath of 99 ± 0.5°C for 100 h or over at oxygen pressure of 0.76 MPa{7.7 kgf/m<sup>2</sup>}.

The inner surface of bomb body, lid and the pipe for attaching pressure gauge shall be finished so as to facilitate washing and cleaning, and the capacity excluding the sample container and the holder shall be 185 ± 6 ml to the contact surface of the gasket of pressure gauge socket. The assembly is shown in Fig. 19.

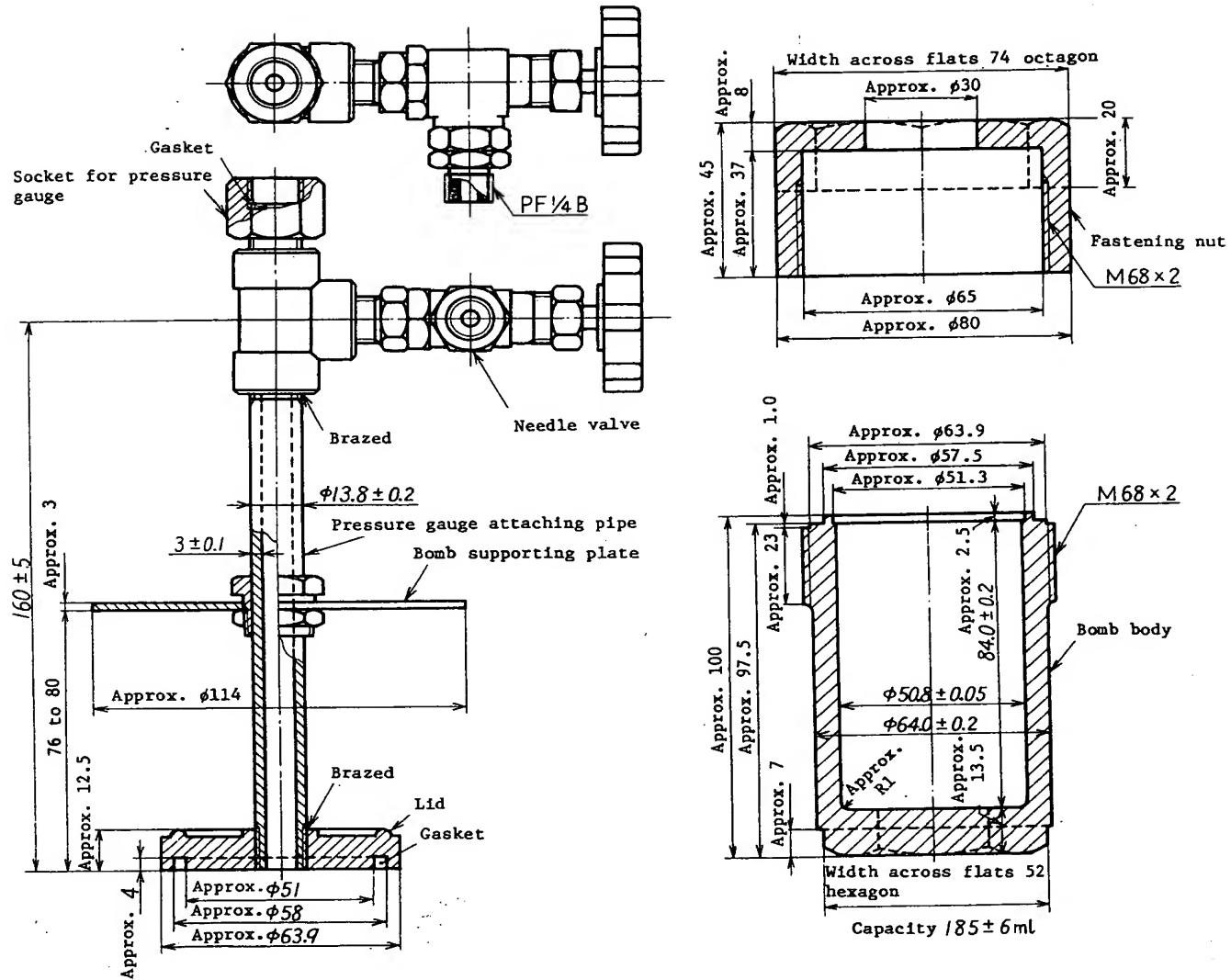
Remarks: The bomb may be of flanged type.

Table 18. Materials of bomb

Name of component	Materials
Bomb body	SUS 304 of JIS G 4303
Fastening nut	Class 3 of JIS H 5101 or C 3602 of JIS H 3250
Socket for pressure gauge	C 3602 of JIS H 3250
Needle valve	SUS 304 of JIS G 4303
Pipe for attaching pressure gauge	SUS 304 TP nominal diameter 1/4 B <sub>5</sub> , nominal thickness schedule 80 of JIS G 3459
Bomb supporting plate	C 2600 P specified in JIS H 3100
Gasket	Nitrile rubber, or synthetic rubber or lead having oil resistance and heat resistance at least equal to nitrile rubber.

Fig. 20. Bomb

Unit: mm



(2) Pressure gauge Pressure gauge (marked as "No Oil")<sup>(16)</sup> of Class 0.5, 150 mm in size, screw joint PF<sub>1/4</sub>, pressure range 0 MPa to 0.98 MPa {0 kgf/cm<sup>2</sup> to 10 kgf/cm<sup>2</sup>} and minimum graduation 9.8 kPa{0.1 kgf/cm<sup>2</sup>}, specified in JIS B 7505 (1980).

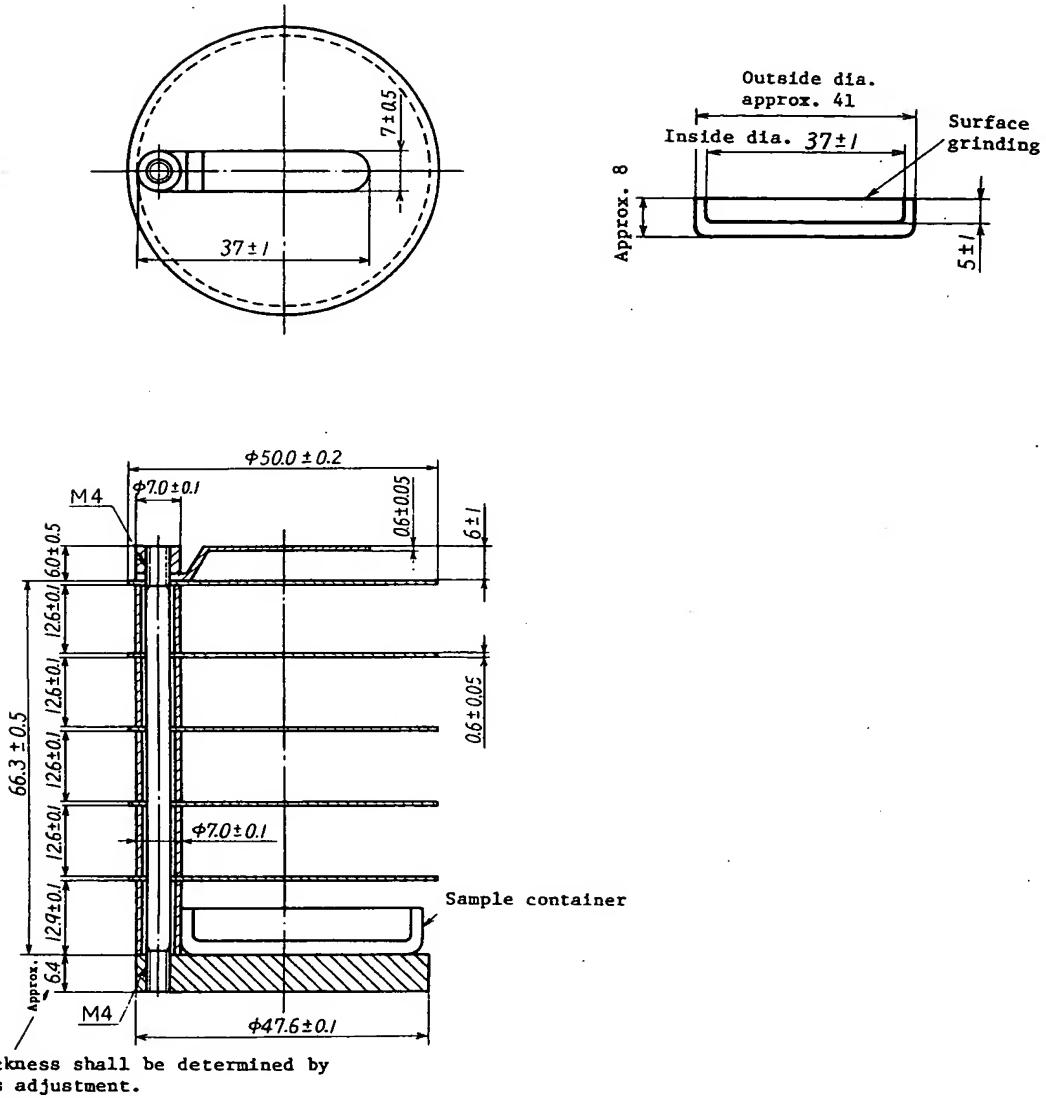
Otherwise, an instrument of indicating or recording type with the same precision may be used.

Note <sup>(16)</sup> For the inspection of pressure gauge, oils shall not be used.

(3) Sample container holder The holder shall have a shape, dimensions and the mass as shown in Fig. 21, and the materials of rod parts shall be SUS 304 of JIS G 4303 and that of plate parts SUS 304 of JIS G 4305.

Fig. 21. Sample container holder and sample container

Unit: mm

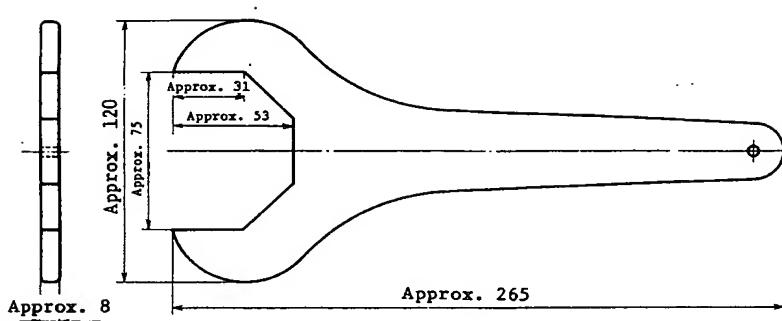


- (4) Sample container The sample container shall be as specified in Fig. 176 of JIS K 2839.
- (5) Fastening tool for bomb Fastening tools are a wrench to fasten the fastening nuts for bomb and a block for fixing the bomb, and their examples are shown in Fig. 22.

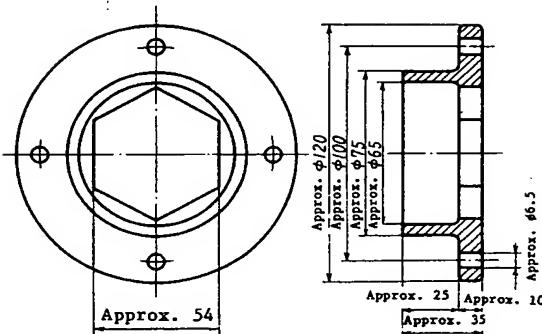
Fig. 22. Fastening tools for bomb (An example)

Unit: mm

A Wrench



B Block for fixing



(6) Oxygen introducing tube Oxygen introducing tube shall be a flexible tube of metal or other appropriate material to connect the oxygen vessel and the bomb for introducing oxygen to the bomb, and shall have metal fittings at both ends to connect the bomb and the oxygen vessel.

(7) Thermostatic oil bath An example of the thermostatic oil bath shall be given in Fig. 19. It shall be equipped with a motor driven agitator, an electric heater and a temperature regulator, and shall be capable of keeping the bath temperature in the range of 99°C to 150°C within  $\pm 0.5^\circ\text{C}$ .

The lid of the bath shall be provided with bomb inserting holes and a thermometer holder. Bomb inserting holes shall be attached with a guide of the supporting plate, and when the bomb is inserted, the distance between the upper surface of the bomb and the level of the bath liquid shall be about 50 mm or over.

Remarks 1. For the liquid of thermostatic bath, it is desirable to use heat resistant silicone oil or the like of having good thermal stability.

2. A safety device for overheating should preferably be attached.

(8) Thermometer The thermometer shall be of No. 6 specified in JIS B 7410.

Informative reference: In the case of testing at over 99°C, other appropriate thermometer shall be used.

5.8.3 Solvents Petroleum benzine specified in JIS K 8594 shall be used.

5.8.4 Preparation of test Preparation of test shall be done as follows:

(1) Wash and clean the sample container with a suitable solvent and after washing with warm soap water, rinse with tap water and then with distilled water thoroughly, and dry in a dryer. Thereafter, the sample container shall not be touched with hand directly.

(2) Wash sufficiently the inner surface of the bomb, the sample container holder, the lid, and the attaching pipe for pressure gauge with petroleum, benzine, and then dry thoroughly.

5.8.5 Test procedures Test procedures shall be as follows:

- (1) Take samples each weighing  $4.00 \pm 0.01$  g in five sample containers so as not to enter air bubbles, make the surface of each sample smooth, and place on the shelves of the sample container holder. When assembling the bomb, stuff loosely rounded glass wool in the bottom of the attaching pipe for pressure gauge.
- (2) Put the sample container holder in the bomb, close with the lid and the fastening nuts. Introduce slowly oxygen specified in JIS K 1101 into the bomb up to the oxygen pressure of  $0.69 \text{ MPa}\{7.0 \text{ kgf/cm}^2\}$  and then release slowly. Repeat the above procedure 4 times. When the pressure reaches  $0.69 \text{ MPa}\{7.0 \text{ kgf/cm}^2\}$  at the fifth charging of oxygen, close the needle valve tightly, and check the presence of the gas leakage either by leaving the bomb quietly for several hours or by immersing the bomb in water.
- (3) After ascertaining that no leakage of the gas exists, put the bomb in the thermostatic oil bath kept at  $99 \pm 0.5^\circ\text{C}$ . Since the pressure of the bomb rises at the beginning of the immersion in the bath, continue the operation of releasing oxygen from time to time for about 2 h so that the pressure may hold stability at  $0.76 \pm 0.005 \text{ MPa}\{7.7 \pm 0.05 \text{ kgf/cm}^2\}$ .
- (4) Read out the decrease of oxygen pressure after 100 h elapse from immersing the bomb in the thermostatic oil bath. Record the pressure every 24 h during the testing period.

5.8.6 Result The average value of two measured results [pressure drop  $\text{MPa}\{\text{kgf/cm}^2\}$ ] obtained in 5.8.5, for the same sample is taken as oxidation stability, which is rounded off to the nearest  $0.005 \text{ MPa}\{0.05 \text{ kgf/cm}^2\}$  in accordance with JIS Z 8401 to be taken as the test result.

5.8.7 Precision<sup>(17)</sup> The precision shall be as follows.

Note <sup>(17)</sup> The precision shall be applicable only to the sample wherein oxygen is absorbed approximately in proportion to time. It should not be applied to the sample wherein the oxygen absorption accelerates rapidly in the midway.

- (1) Repeatability The tolerance on the difference between two successive test results, obtained by the same person with the same apparatus in the same laboratory on identical test sample shall be as given in Table 19.

Table 19. Repeatability

Pressure drop (MPa){kgf/cm <sup>2</sup> }	Tolerance (MPa){kgf/cm <sup>2</sup> }
Under 0.034{0.35}	0.015{0.15}
0.034{0.35} to 0.069{0.70} excl.	0.029{0.30}
0.069{0.70} to 0.137{1.40} excl.	0.039{0.40}
0.137{1.40} to 0.378{3.85} excl.	0.069{0.70}

(2) Reproducibility The tolerance on the difference between two single and independent test results, obtained by different persons with different apparatuses in different two laboratories on identical test sample shall be as given in Table 20.

Table 20. Reproducibility

Pressure drop (MPa){kgf/cm <sup>2</sup> }	Tolerance (MPa){kgf/cm <sup>2</sup> }
Under 0.034{0.35}	0.039{0.40}
0.034{0.35} to 0.069{0.70} excl.	0.054{0.55}
0.069{0.70} to 0.137{1.40} excl.	0.083{0.85}
0.137{1.40} to 0.378{3.85} excl.	0.137{1.40}

Remarks: The test result exceeds the tolerance shall be treated in accordance with JIS Z 8402.

### 5.9 Test method for deleterious particles

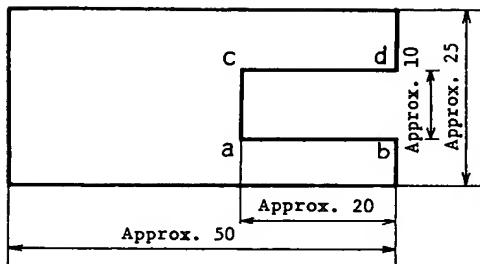
5.9.1 Summary of test method Fill the notch of the specified templet with sample in a clean environment, and then measure the number of the deleterious particles contained in the sample according to the size by using a microscope.

5.9.2 Apparatus and device The apparatus and device shall be composed of the following items (1) to (4).

- (1) Microscope The microscope of about 100 magnifications provided with an ocular micrometer and a mechanical stage.
- (2) Templet The templet shall be a metallic plate with  $0.1 \pm 0.01$  mm thickness of the shape and dimensions as shown in Fig. 23, which shall have a notch of about 10 mm in width and about 20 mm in length.

Fig. 23. Templet

Unit: mm



- (3) Desiccator The desiccator shall have a size enough to contain slide glasses for microscope, and shall be capable of obtaining reduced pressure by the aid of a vacuum pump.
- (4) Vacuum pump The vacuum pump shall be able to reduce rapidly the pressure of air in the desiccator to 1.33 kPa{10 mmHg} or under.

#### 5.9.3 Test procedures Test procedures shall be as follows:

- (1) Remove the surface of the sample with a spatula, place the templet on the slide glass, and fill the notch with the sample<sup>(18)</sup> slightly over the upper surface of the templet while pressing the end without notch by hand.

Note (18) If necessary, air shall be removed by pretreatment.

- (2) When air bubbles are contained in the grease existing in the notch of the templet, put the sample together with slide glass in the desiccator to subject to the pressure reduction treatment, which is made by keeping pressure 1.33 kPa{10 mmHg} or under for 10 min to 15 min. Slide the cover glass while pressing it to the templet so that the excess sample be scraped off from the edge of the notched side of the templet.
- (3) Place the slide glass on the stage of the microscope, and adjust the ocular lens and stage so that the graduation of the micrometer comes to line a-b of the notch.
- (4) Move the stage along the line a-c of the notch while focussing on the deleterious particles, record the numbers of deleterious particles crossing the graduation of the ocular micrometer while classifying into four classes of, 10  $\mu\text{m}$  or over to and excluding 25  $\mu\text{m}$ , 25  $\mu\text{m}$  or over to and excluding 75  $\mu\text{m}$ , 75  $\mu\text{m}$  or over to and excluding 125  $\mu\text{m}$ , and 125  $\mu\text{m}$  or over, continue the measurement until the graduation arrives at the line c-d of the notch of the templet, and take this classification as first section. In this case, for a fibrous matter measurement shall be made for the width, not for the length.

Remarks: Particles 10  $\mu\text{m}$  or smaller shall not be counted.

(5) Shift the stage along the line c-d of the notch adjacent to the first section by equal distance of the graduation length of micrometer, and count and record the numbers of particles by the method specified in (4) until the stage reaches the line a-c of the notch. Repeat the procedure to about 10 mm on the line a-b or c-d of the notch.

**5.9.4 Calculation and result** Calculate the number of deleterious particles in the sample per 1 cm<sup>3</sup> by the formula below, and express the number of particles in an average for every size obtained to digit of integer from three test results by rounding off the value according to JIS Z 8401.

$$A' = \frac{1000 (A+B+C+D)}{T \cdot S \cdot N}$$

$$B' = \frac{1000 (B+C+D)}{T \cdot S \cdot N}$$

$$C' = \frac{1000 (C+D)}{T \cdot S \cdot N}$$

$$D' = \frac{1000 D}{T \cdot S \cdot N}$$

where, A' : the number of particles of 10  $\mu\text{m}$  or over in the maximum size per 1 cm<sup>3</sup> sample (particles/cm<sup>3</sup>)

B' : the number of particles of 25  $\mu\text{m}$  or over in the maximum size per 1 cm<sup>3</sup> sample (particles/cm<sup>3</sup>)

C' : the number of particles of 75  $\mu\text{m}$  or over in the maximum size per 1 cm<sup>3</sup> sample (particles/cm<sup>3</sup>)

D' : the number of particles of 125  $\mu\text{m}$  or over in the maximum size per 1 cm<sup>3</sup> sample (particles/cm<sup>3</sup>)

A : total number of particles of 10  $\mu\text{m}$  or over to and excl. 25  $\mu\text{m}$  (particles)

B : total number of particles of 25  $\mu\text{m}$  or over to and excl. 75  $\mu\text{m}$  (particles)

C : total number of particles of 75  $\mu\text{m}$  or over to and excl. 125  $\mu\text{m}$  (particles)

D : total number of particles of 125  $\mu\text{m}$  or over (particles)

T : sectional area of notch of templet (mm<sup>2</sup>)<sup>(19)</sup>

S : length of graduation of ocular micrometer (mm)

N : the number of measuring sections

Note <sup>(19)</sup> Measure the thickness of templet and the width of the notch to calculate the sectional area (mm<sup>2</sup>).

## 5.10 Test method for ash content

**5.10.1 Summary of test method** After burning the sample until carbon disappears, weigh the residue to obtain the ash content.

**5.10.2 Apparatus and device** The apparatus and device shall be composed of the following (1) and (2).

(1) Crucible The crucible shall be of 15 ml capacity, and made of porcelain, quartz or platinum<sup>(20)</sup>.

Note <sup>(20)</sup> When a sample contains lead, zinc and other substances which react with platinum at high temperature, platinum crucible shall not be used.

(2) Muffle furnace A muffle furnace of appropriate size and capable of maintaining the temperature up to 600°C.

#### 5.10.3 Test procedures Test procedures shall be as follows:

Heat a crucible to red, and weigh after cooling in a desiccator. Take 2 g to 5 g of sample into this crucible, and weigh to the nearest 0.01 g. Heat the crucible, and burn it slowly<sup>(21)</sup>). After burning, further ignite strongly in the muffle furnace until the carbon disappears. Leave the crucible and the content still in a desiccator for cooling, and weigh.

Note <sup>(21)</sup> When a sample scatters by foaming, add 1 ml to 2 ml of ethyl alcohol before heating.

#### 5.10.4 Calculation and result Calculate ash content according to the following formula. Round off the average of two measured results of the same sample obtained in 5.10.3 to one place of decimal in accordance with JIS Z 8401 to be taken as the test result.

$$A = \frac{W_r}{W_s} \times 100$$

where,  $A$  : ash content (mass %)

$W_r$  : mass of ash (g)

$W_s$  : mass of sample (g)

#### 5.11 Test method for worked stability

##### 5.11.1 Summary of test method After working the sample one hundred thousand times in the specified worker, keep it at 25°C, further work 60 times, and measure the penetration.

##### 5.11.2 Tester for worked stability The tester for worked stability shall consist of (1) to (6) below.

(1) Worker for worked stability The worker is exemplified in Fig. 24, and capable of moving up and down the perforated plate attached to the top of the sliding rod.

The gland of the sliding rod and the jointing part of the cup and the cover or the like shall be so constructed that the sample in the cup should leak extremely little for one hundred thousand up and down double strokes.

(2) Motor driven working apparatus The apparatus shall be similar to the motor driven working apparatus specified in 5.3.2 (6) in construction and shall be capable of moving up and down perforated plate of the worker for the worked stability at a rate of  $60 \pm 10$  double strokes per 1 min for 67 mm to 69 mm travel. The mechanism of up and down movement shall be capable of withstanding the test for worked stability, and it is preferable that the motor to be used is of 0.75 kW.

(3) Penetrometer As specified in 5.3.2 (1).

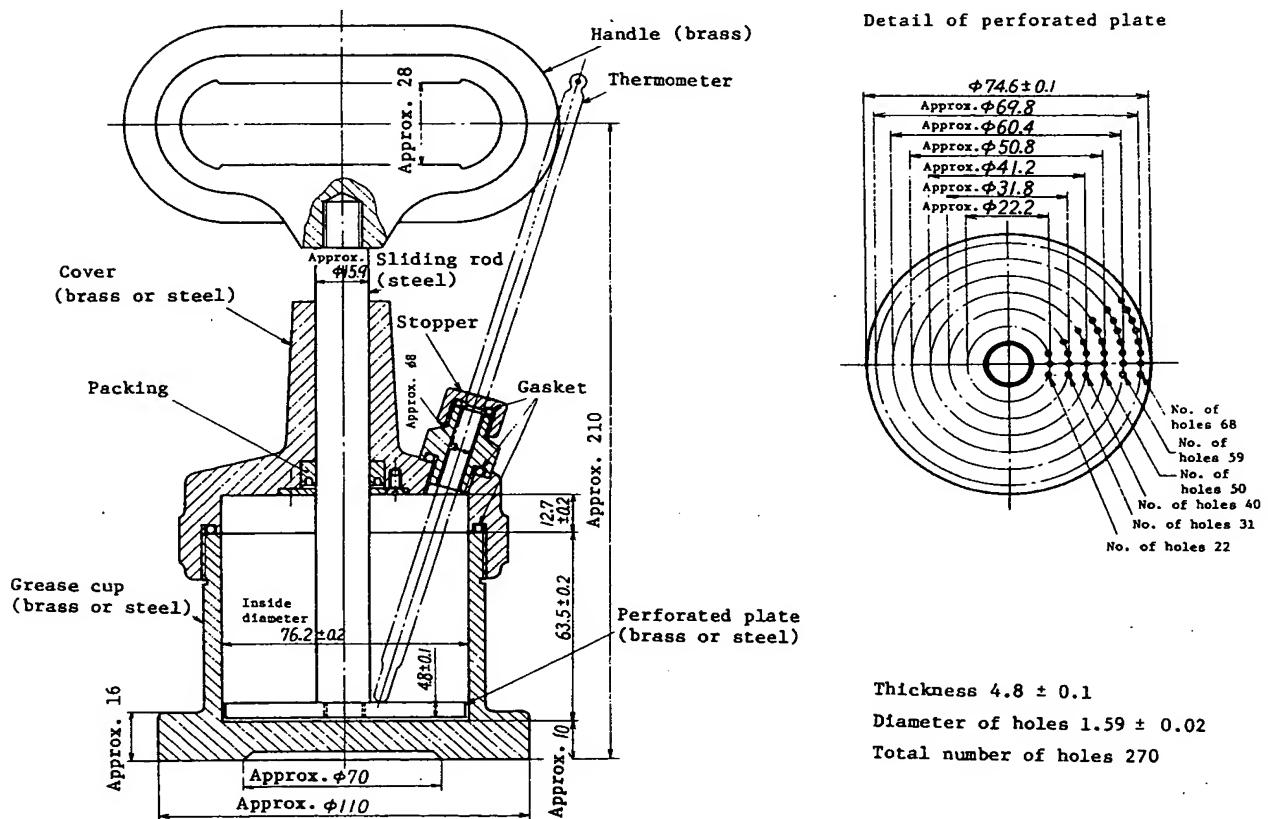
(4) Cone As specified in 5.3.2 (2) and (3).

(5) Spatula As specified in 5.3.2 (7).

(6) Thermostatic water bath As specified in 5.3.2 (9).

Fig. 24. Worker for worked stability (An example)

Unit: mm



### 5.11.3 Test procedures Test procedures shall be as follows:

(1) Prepare about 500 g of sample which is enough to fill the cup of the worker, and leave it still until it coincides with room temperature of the testing place.

Remarks: The temperature of the testing room should preferably be kept at 15°C to 30°C.

- (2) After leaving the sample for enough time in the testing place, fill the cup of clean grease worker with sample with a spatula with care not to allow air to enter the sample, and mound it to a mountain shape so that the central part will be about 10 mm or higher than the rim of the cup, open the cock on the cover of the grease worker, and assemble the grease worker. Then depress the perforated plate to the bottom of the cup, close the cock, fit the worker to the motor driven working apparatus, and work the sample until it reaches one hundred thousand times continuously (about 28 h).
- (3) After working, immediately remove the grease worker from the motor driven working apparatus, leave it in the thermostatic water bath kept at  $25 \pm 0.5^{\circ}\text{C}$  for 2 h, and prepare the sample by the method specified in 5.3.3 (1)(b) and (c). In this case, the perforated plate as specified in 5.11.2 shall be used.
- (4) Measurement of the penetration of the sample shall be made according to the method specified in 5.3.4 (2).

5.11.4 Result The average of three test results obtained in 5.11.3 for the same sample shall be taken as worked stability, and rounded off to an integer in accordance with JIS Z 8401 to be taken as the test result.

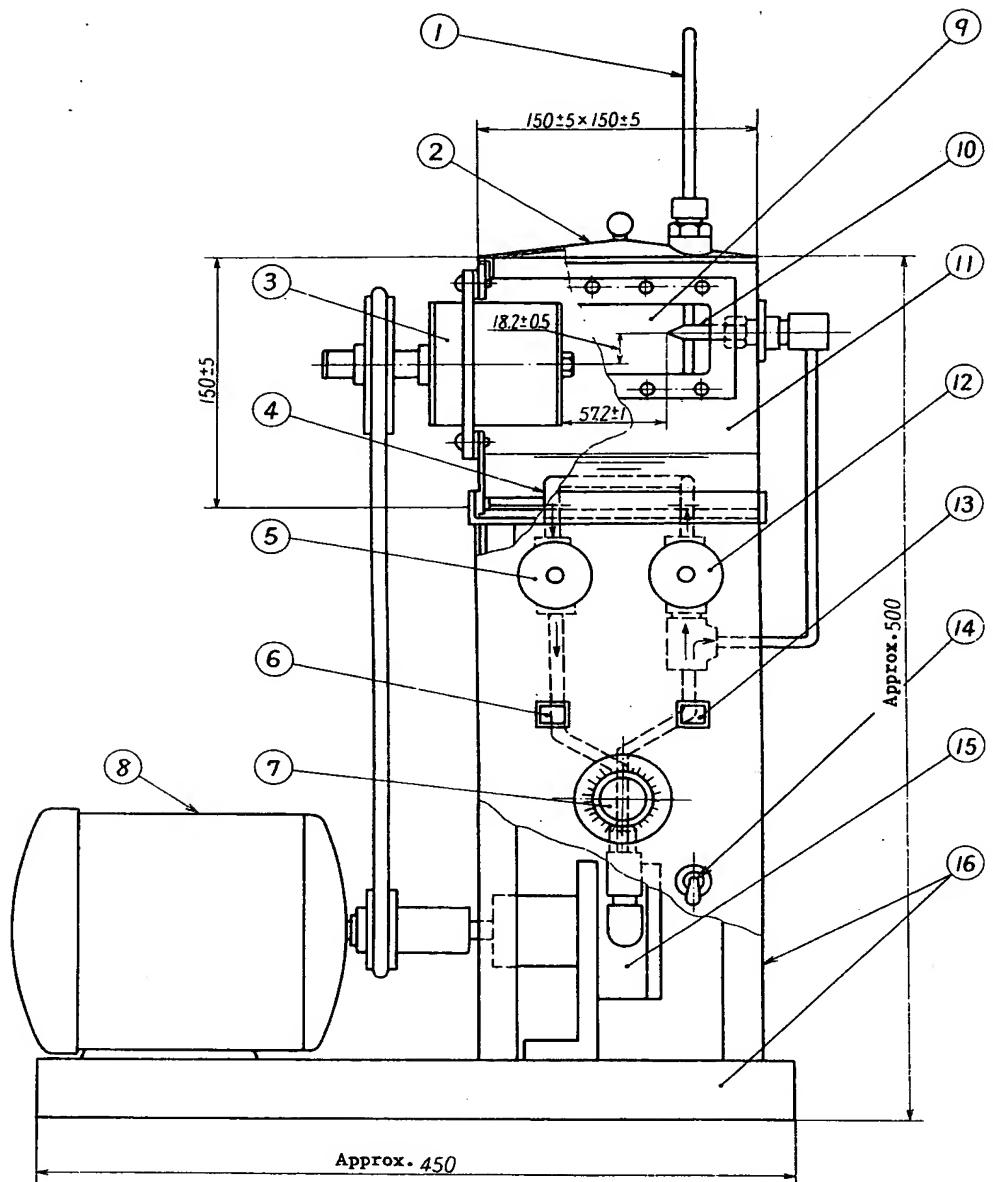
## 5.12 Test method for water washout resistance

5.12.1 Summary of test method Fit the ball bearing having been packed with the sample in a housing, and rotate at a rate of  $600 \pm 30$  revolutions per min, and jet distilled water kept at  $38 \pm 2^{\circ}\text{C}$  or  $79 \pm 2^{\circ}\text{C}$  to the ball bearing housing at the rate of  $5 \pm 0.5$  ml per s. Obtain the loss of the sample (mass %) after 1 h lapse.

5.12.2 Testing apparatus for water washout resistance The testing apparatus for water washout resistance shall be composed of the following items (1) to (4). An example of the construction of testing apparatus for water washout resistance is given in Fig. 25.

Fig. 25. Construction of testing apparatus for water washout resistance (An example)

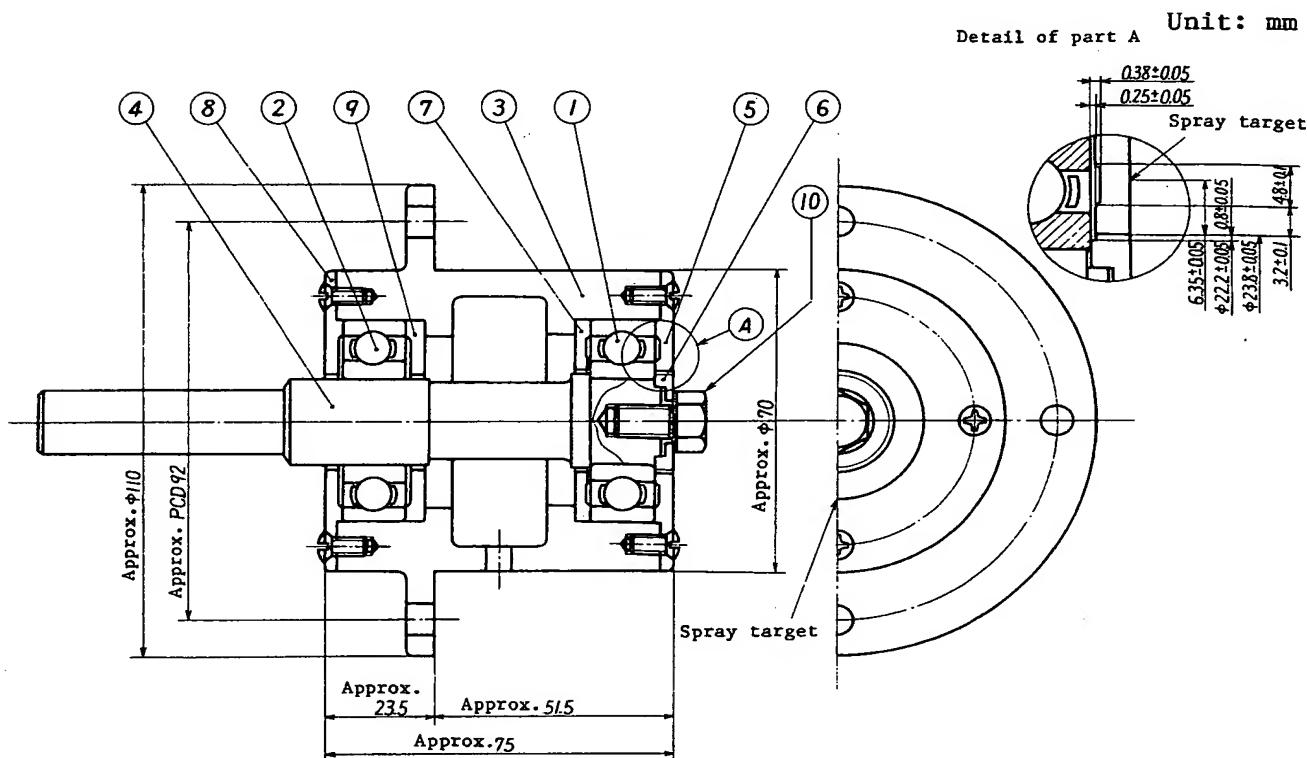
Unit: mm



① Thermometer	⑨ Peep window
② Lid	⑩ Spray nozzle
③ Housing	⑪ Thermostatic water bath
④ Electric heater	⑫ By-pass valve (for regulating the amount of spray)
⑤ Flow regulating valve	⑬ Indicator lamp of power source
⑥ Signal lamp of temperature regulation	⑭ Switch of power source
⑦ Temperature regulator	⑮ Pump
⑧ Motor	⑯ Frame

(1) Rotating mechanism of ball bearing for test The mechanism is to rotate the ball bearing contained in the housing shown in Fig. 26 at a rate of  $600 \pm 30$  revolutions per min, and shall consist of the housing, the ball bearing for test and a suitable driving mechanism.

Fig. 26. Construction of housing and shaft (An example)



① Ball bearing for test	⑦ Outer ring holder of ball bearing for test
② Ball bearing for supporting	⑧ Outer ring retainer of ball bearing for supporting
③ Housing	⑨ Outer ring holder of ball bearing for supporting
④ Shaft	⑩ Fastening bolt
⑤ Outer ring retainer of ball bearing for test	
⑥ Inner ring retainer of ball bearing for test	

(a) Ball bearing for test Tolerance class 0, 6204 open type specified in JIS B 1521.

(b) Housing and shaft The housing and the shaft shall be as shown in Fig. 26 in shape and dimensions, made of brass or stainless steel (SUS304), and capable of being attached or detached easily to the thermostatic water bath.

(2) Thermostatic water bath The water bath shall be as shown in Fig. 25 in shape and dimensions and equipped with the electric heater, temperature regulator, suitable lid and frame, capable of keeping water temperature at  $38 \pm 2^\circ\text{C}$  or  $79 \pm 2^\circ\text{C}$ . The bath shall be easily attached with the housing and spray nozzle at the position shown in Fig. 25.

(3) Water spray mechanism The mechanism shall consist of the spray nozzle, by-pass valve, flow-rate regulating valve, pump, motor and others, and shall be capable of circulating and spray the warm water in the bath to the housing at a rate of  $5 \pm 0.5 \text{ ml per s}$ .

The test for the spray flow rate shall be made by measuring the flow quantity for 60 s or 30 s after jointing a rubber tube to the top end of the spray nozzle and putting the other end of the rubber tube to a receiving measuring cylinder. The spray flow rate of the spray nozzle shall be regulated by the operation of flow-rate regulating valve and the by-pass valve.

(a) Spray nozzle The nozzle shall be  $1.0 \pm 0.1 \text{ mm}$  in inner diameter, and when attached to the thermostatic water bath, the spray shall be able to impinge on the spray target specified in Fig. 26 without spreading.

(b) Pump The pump shall be capable of spray the warm water from the spray nozzle without pulsations at the specified flow rate.

(4) Thermometer The thermometer shall be a glass thermometer which is readable at  $38^\circ\text{C}$  and  $79^\circ\text{C}$  with scale interval of  $1^\circ\text{C}$  or under, or a thermometer at least equal in quality thereto.

5.12.3 Thermostatic air bath That shall be capable of keeping the specified temperature within  $\pm 1^\circ\text{C}$  by electric heater.

5.12.4 Reagent Solvent shall be petroleum benzine specified in JIS K 8594.

5.12.5 Preparation of test Preparation of test shall be carried out as follows:

(1) Clean the water bath and the water circulating passages with distilled water. Wipe off any oil scum adhering on to the inner wall of the bath.

(2) Clean the ball bearing for test with petroleum benzine, and dry.

5.12.6 Test procedures Test procedures shall be as follows:

(1) Pack the ball bearing for test of known mass with  $4.00 \pm 0.05 \text{ g}$  of sample. Attach this bearing and outer ring retainer (Fig. 26 (5)), outer ring holder (Fig. 26 (7)) and inner ring retainer (Fig. 26 (6)) of ball bearing for test of known mass into the housing, as in Fig. 26.

(2) Add minimum 750 ml of distilled water in the bath<sup>(22)</sup>, start heating and driving the motor. At this time, care shall be taken so that diverting water spray is introduced into water with a rubber tube connected to the tip of the spray nozzle to prevent the housing from being wetted, and adjustment is made so as to keep the water temperature at  $38 \pm 2^\circ\text{C}$  or  $79 \pm 2^\circ\text{C}$ .

- (3) After the adjustment of water temperature, adjust to keep the water flow rate at  $5 \pm 0.5$  ml per s by putting the end of rubber tube in a measuring cylinder.
- (4) Detach the tube from the nozzle, and control the water spray so as to impinge on the spray target 6 mm above the clearance between the outer ring retainer and the inner ring retainer. Rotate the ball bearing for test at  $600 \pm 30$  revolutions per min for 1 h.
- (5) Detach the test ball bearing, outer ring holder, outer ring retainer and inner ring retainer from the housing, and place them on a watch glass of known mass, and dry in the thermostatic air bath kept at  $77 \pm 1^\circ\text{C}$  for 16 h. At this time, separate outer ring holder, outer ring retainer and inner ring retainer from the bearing, and place with their inner surfaces upward on the watch glass.
- (6) After drying, leave in a desiccator for cooling to room temperature, measure the mass of the test ball bearing, outer ring holder, outer ring retainer, inner ring retainer and watch glass to the nearest 0.01, and obtain the loss in mass of the sample(<sup>23</sup>).

Notes (<sup>22</sup>) The level of the distilled water shall be lower than the bottom end of the housing.

(<sup>23</sup>) The sample adhering onto the outer ring holder, outer ring retainer, inner ring retainer and the watch glass shall not be considered to be the loss in mass.

**5.12.7 Calculation and result** Calculate the water washout resistance of grease from loss (mass %) of sample according to the following formula. The value shall be rounded off to one place of decimal in accordance with JIS Z 8401 to take it as test result.

$$A = \frac{C}{B} \times 100$$

where, A : water washout resistance (mass %)

C : loss in mass of sample (g)

B : mass of sample (g)

**5.12.8 Precision** The tolerance on the difference between the results of two tests made for the same sample, once respectively by different persons with different apparatus in two different testing rooms shall be given as follows:

Tolerance at $38^\circ\text{C}$	20 % of average value
79°C	25 % of average value

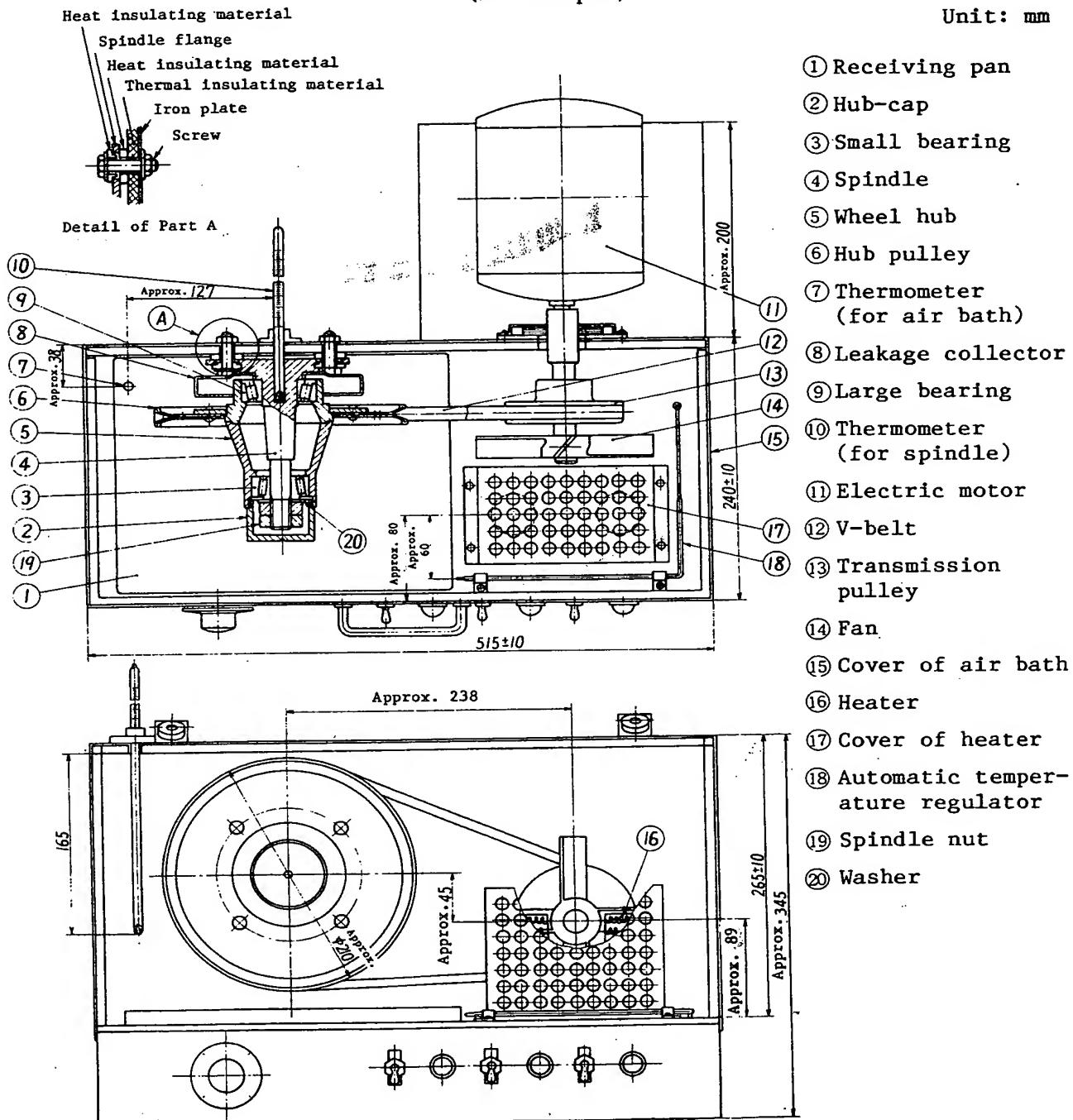
### **5.13 Test method for leakage tendency of grease**

**5.13.1 Summary of test method** Fill sample grease of specified quantity in the wheel-hub and the bearing, rotate under the specified conditions, and measure the total mass of the leaked grease and oil to take it as leakage tendency.

Further, examine the presence of substance adhering onto bearing surface.

5.13.2 Testing apparatus for leakage tendency The testing apparatus for leakage tendency shall be composed of the following items (1) to (7). An example of the construction of the testing apparatus for leakage tendency is given in Fig. 27.

Fig. 27. Construction of testing apparatus for leakage tendency  
(An example)



(1) Thermostatic air bath The air bath shall be as shown in Fig. 27 in shape and dimensions, equipped with a heater capable of maintaining the bath temperature at  $113 \pm 2^\circ\text{C}$ , a temperature regulator and a fan. The heater shall be capable of raising the bath temperature to  $113^\circ\text{C}$  within  $15 \pm 5$  min. The bath contains the rotating mechanism of wheel bearing specified in (2) below.

(2) Rotating mechanism of wheel bearing The mechanism is to rotate the wheel hub at a rate of  $660 \pm 30$  revolutions per min, and shall consist of the spindle, wheel hub, bearing, hub pulley transmission pulley, V-belt, electric motor and the like as shown in Fig. 27.

(a) Spindle and wheel hub The spindle and the wheel hub shall be as shown in Fig. 28 and Fig. 29 in shapes and dimensions, and the materials shall be as specified below.

Spindle: As specified in JIS G 4105.

Wheel hub: As specified in JIS G 4051.

Fig. 28. Spindle (An example)

Unit: mm

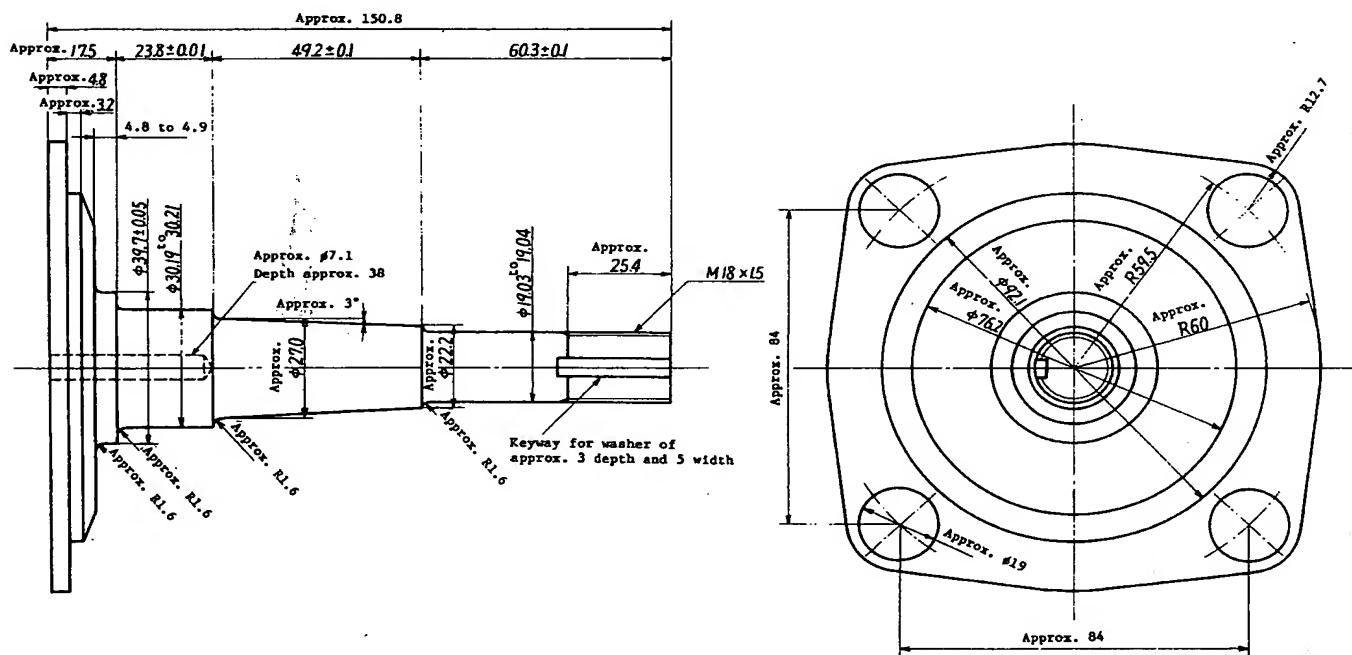
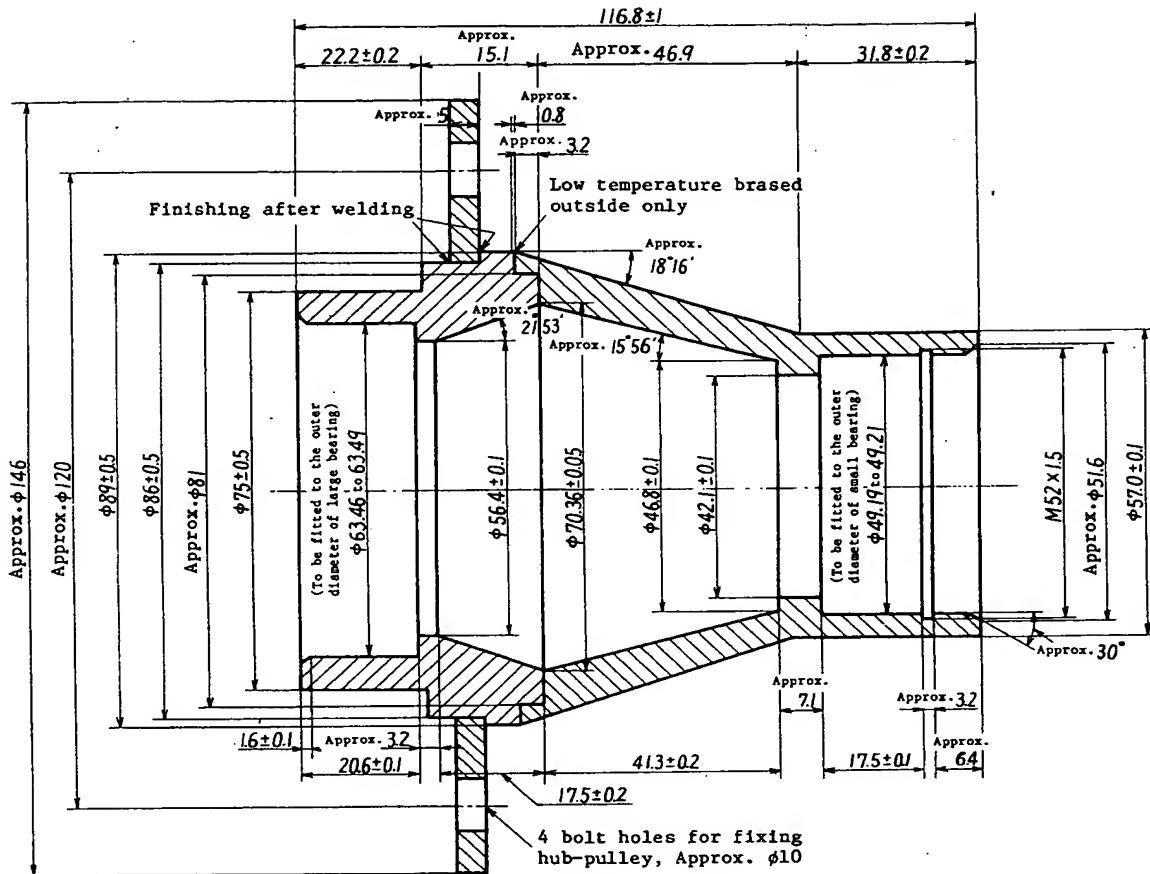


Fig. 29. Wheel hub (An example)

Unit: mm



Remarks: Polish inside tapered surfaces with P100 or coarser garnet abrasive paper specified in JIS R 6252. However, do not round off sharp edges when polishing.

(b) Bearing Two types of tapered roller bearings shown in Table 21 shall be used.

Table 21. Tapered roller bearing

Unit: mm

	Bearing bore diameter	Bearing outside diameter	Assembled bearing width	Inner ring width	Outer ring width
Large bearing	30.213	63.500	20.638	20.638	15.875
Small bearing	19.050	49.225	23.020	21.539	17.463

**Informative reference:** The large bearing corresponds to 15118/15250 X in Timken's nominal number, the small bearing to 09074/09196 in Timken's nominal number.

(c) Hub pulley and transmission pulley The hub pulley shall be as shown in Fig. 30 in shape and dimensions, made of steel, and shall be fixed to the wheel hub by bolts, rivets or others. The transmission pulley shall be a metallic pulley capable of rotating hub pulley at a rate of  $660 \pm 30$  revolutions per min.

(d) Motor and belt The motor shall be to drive the wheel hub and the fan in thermostatic air bath, and should preferably be about 0.2 kW in capacity. The belt shall be Type A specified in JIS K 6323.

(e) Fan The fan shall be as shown in Fig. 31 in shape and dimensions, made of light alloy, and shall be jointed directly to the motor shaft, to be driven to the direction of blasting the heater.

Fig. 30. Hub pulley (An example)

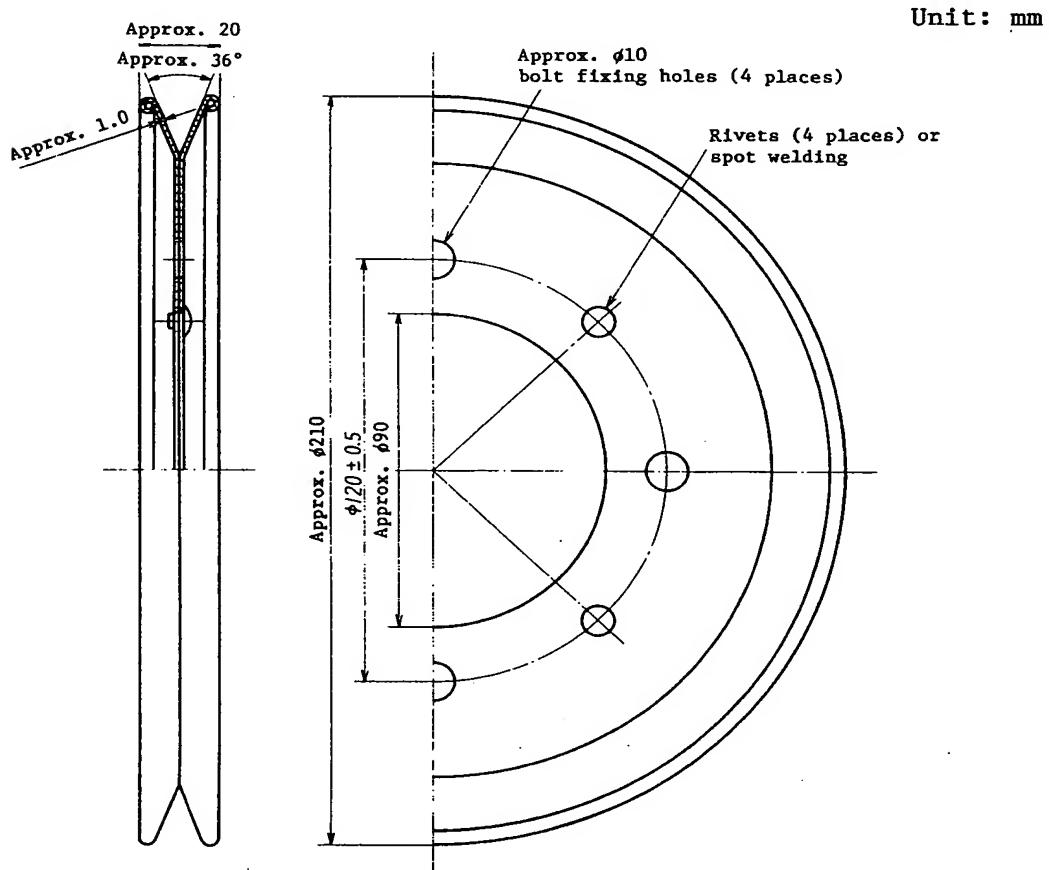
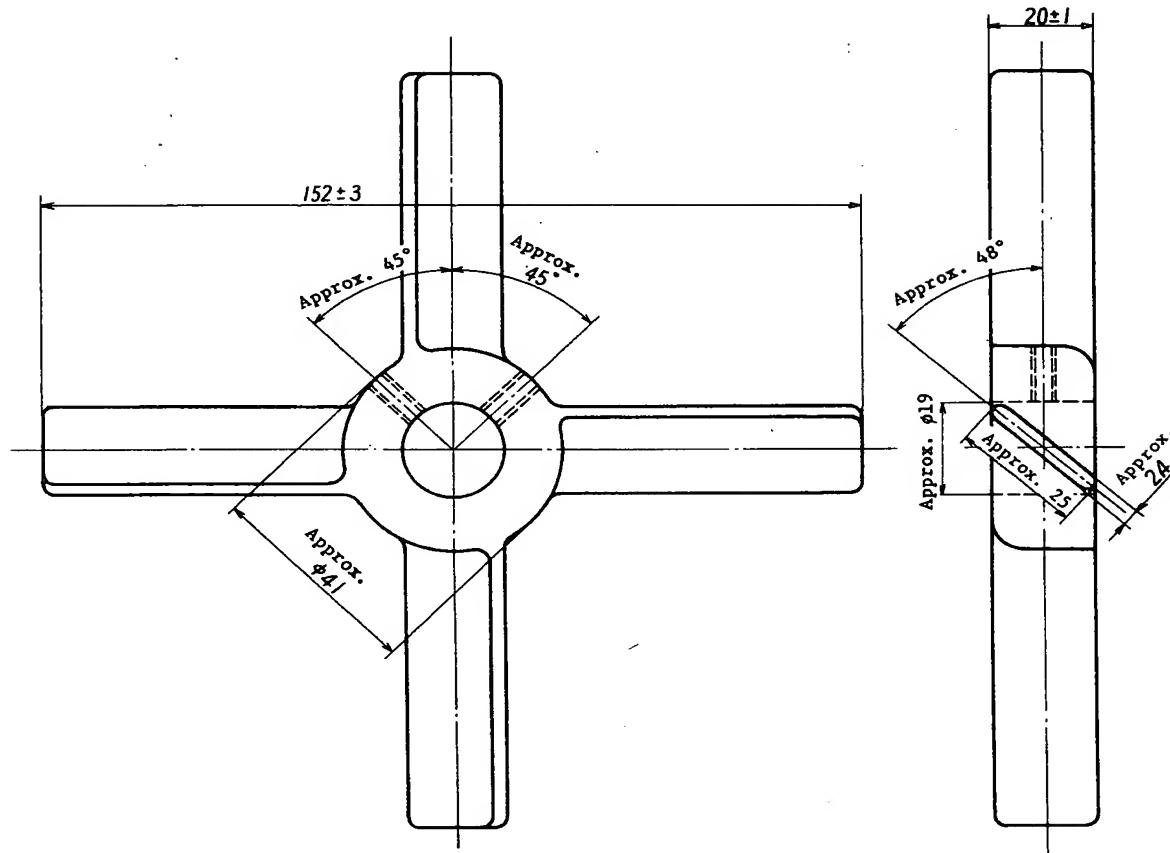


Fig. 31. Fan (An example)

Unit: mm



(3) Hub cap The hub cap shall be made of metal into the shape and dimensions shown in Fig. 32.

(4) Leakage collector The collector shall be made of light alloy into the shape and dimensions shown in Fig. 33.

Fig. 32. Hub cap (An example)

Unit: mm

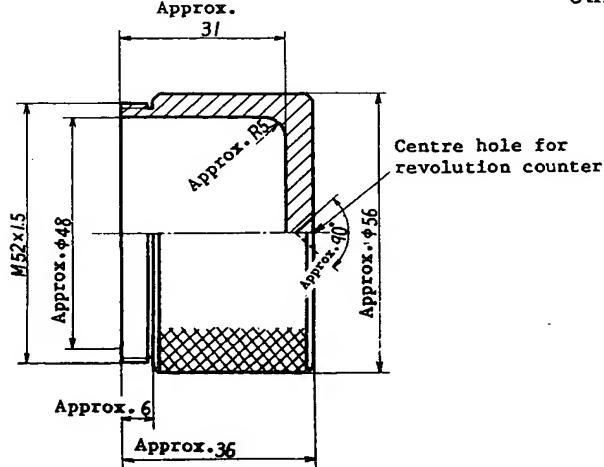
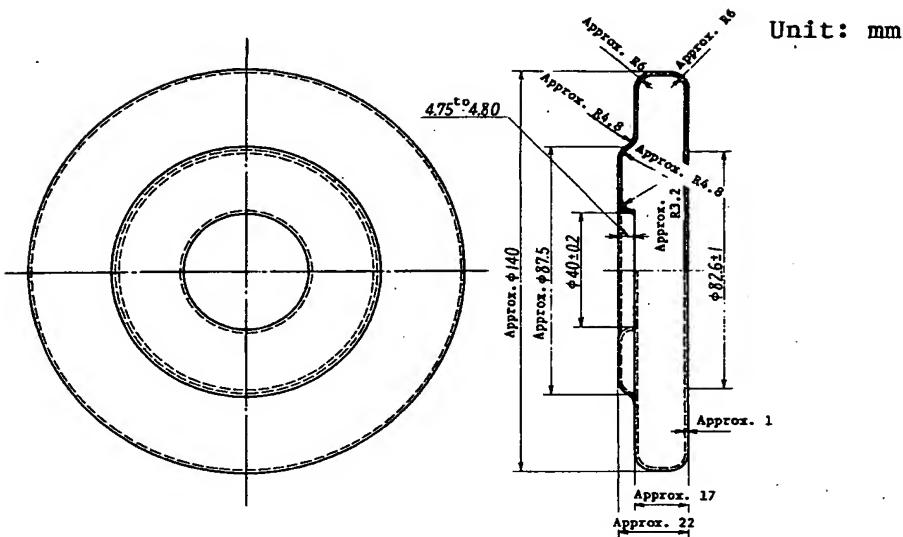


Fig. 33. Leakage collector (An example)



- (5) Receiving pan The pan shall be a rectangular pan made of stainless steel and the dimensions of about 220 mm in length, about 280 mm in breadth and about 13 mm in depth may be suitable.
- (6) Torque wrench The wrench shall be capable of fastening the spindle nut shown in Fig. 27 with a torque of  $6.8 \pm 0.2 \text{ N}\cdot\text{m}$  { $69 \pm 2 \text{ kgf}\cdot\text{cm}$ }.
- (7) Thermometer The thermometer shall be No. 34 specified in JIS B 7410.

5.13.3 Reagent The solvent shall be petroleum benzine specified in JIS K 8594.

5.13.4 Preparation of test The preparation of test shall be as follows:

- (1) Weigh out  $90 \pm 1$  g of sample on a watch glass, pack with a narrow wedge-shape spatula  $2.0 \pm 0.1$  g of grease in the small bearing and  $3.0 \pm 0.1$  g in the large bearing<sup>(24)</sup>. Distribute the remaining test sample grease (about 85 g) to a uniform layer on the inside of the wheel hub, and coat a thin film of sample over the large and small bearing outer ring raceway surfaces<sup>(25)</sup>.

Notes (24) In place of a spatula, 10 ml to 20 ml glass syringe specified in JIS T 3201 may be used.

(25) For packing the remaining sample grease in the wheel hub, use a spatula about 150 mm long so as to make the surfaces of the sample come to the same height with those of the outer ring raceway of the large and small bearings.

In the case where further excess sample remains, pack the remaining sample so as to mound at the centre of the wheel hub.

(2) Weigh the leakage collector, the hub cap and the receiving pan each to the nearest 0.1 g, attach the leakage collector to the fixed position of the spindle, mount the large bearing to the spindle, and then mount the wheel hub and the small bearing with care not to allow the packed sample to contact the spindle. Then put a washer at the top of the small bearing, fit the first hexagon nut, fasten with a force of  $6.8 \pm 0.2 \text{ N}\cdot\text{m}$  { $69 \pm 2 \text{ kgf}\cdot\text{cm}$ } with a torque wrench, return the nut by an angle of  $60 \pm 5^\circ$ , and fix the first hexagon nut by fastening the second hexagon nut.

Remarks: The bearings, the wheel hub and the spindle shall be examined thoroughly at every test, and it shall be ascertained that they have no abrasion and other defects.

The use of bearings shall not exceed 250 test times.

(3) After fixing the hexagon nut to the spindle, mount the hub cap to the wheel hub, put the V-belt around the pulley precisely<sup>(26)</sup>, place the receiving pan at the fixed place and close the cover of air bath.

Note (26) In order to avoid the dispersion of the test results, ascertain that the transmission pulley and hub pulley are aligned in a straight line.

#### 5.13.5 Test procedures Test procedures shall be as follows:

(1) After the completion of the preparation of test in accordance with 5.13.4, switch on the motor and the heater to rotate the wheel hub at a rate of  $660 \pm 30$  revolutions per min, raise the temperature of the air bath to  $113 \pm 3^\circ\text{C}$  within  $15 \pm 5$  min reckoning from the starting of the test to let the temperature of the spindle arrive at  $104 \pm 1.5^\circ\text{C}$  within  $60 \pm 10$  min, and maintain the spindle temperature to the finish of the test.

(2) Immediately after 6 h elapse, switch off the motor and the electric heater, open the cover of air bath, detach hub cup, large and small bearings, wheel hub and leakage collector from the spindle, leave the leakage collector and the hub cap still to cool down to room temperature, measure the mass to the nearest 0.1 g.

When the sample grease floods over the leakage collector to deposit in the receiving pan, measure the mass of the receiving pan to the nearest 0.1 g.

(3) Immerse the large and small bearings which have been detached from the spindle in accordance with (2) above in a beaker containing petroleum benzine at room temperature for 2 min to remove the sample grease adhering onto the bearings, and examine the presence of varnish, gum or lacquer-like deposits on the surfaces.

#### 5.13.6 Result Take the total mass of leaked sample grease in the leakage collector and the hub cap measured to the nearest 0.1 g as the leakage tendency.

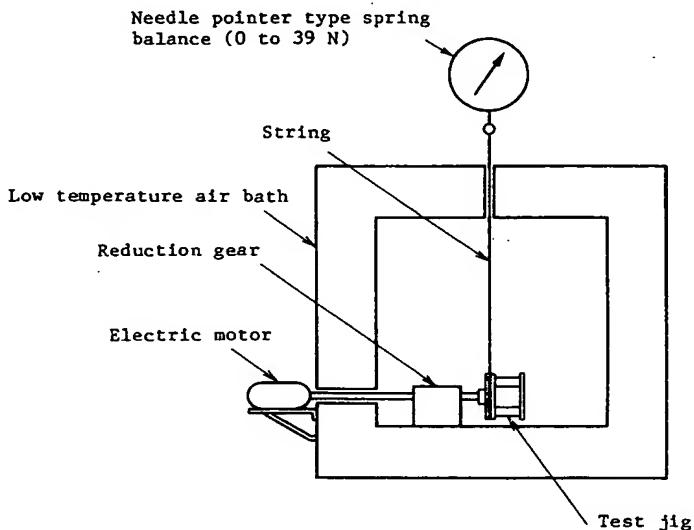
When the sample deposits in the receiving pan, add the mass to the leakage tendency defined above. In this case, whether or not varnish, gum or lacquer-like deposits present on the surface of the bearings shall be appended to the test report.

### 5.14 Test method for low temperature torque

5.14.1 Summary of test method Fill an open type designation 6204 ball bearing specified in JIS B 1521 with sample grease, chill the ball bearing to the specified temperature, and further maintain this temperature for 2 h. Then rotate the inner ring of the ball bearing at a rate of one revolution per min, and measure the restraining force (torque) of the outer ring.

5.14.2 Testing apparatus for low temperature torque The apparatus shall consist of the following parts (1) to (6). An example of the construction of the testing apparatus for low temperature torque is given in Fig. 34.

Fig. 34. Construction of testing apparatus for low temperature torque (An example) (One sample mounting)



(1) Low temperature air bath A bath of  $0.03 \text{ m}^3$  or more in inner capacity, capable of controlling the temperature in the bath at the specified temperature within  $\pm 1^\circ\text{C}$ <sup>(27)</sup>. The driving mechanism may be mounted externally<sup>(28)</sup> or directly in the bath.

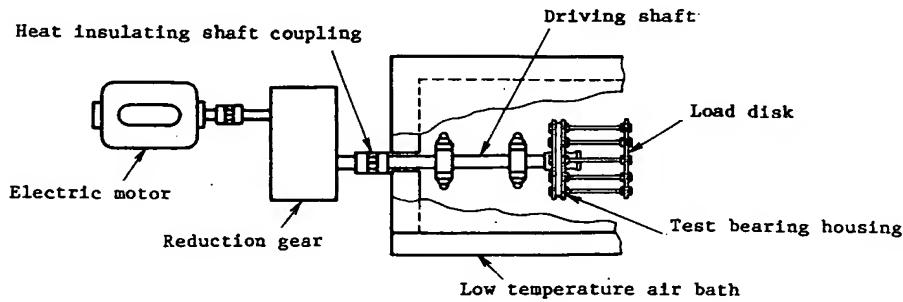
Notes <sup>(27)</sup> A partition wall shall be interposed to avoid the direct radiation from the test bearing to the cooling medium bath.

<sup>(28)</sup> When the driving mechanism is provided outside, the temperature on the surface of the driving shaft between the test bearing and inner wall of the low temperature air bath shall not be  $1^\circ\text{C}$  or higher than the specified temperature.

(2) Bearing Ball bearing for the test shall be open type designation 6204 ball bearing specified in JIS B 1521, of which tolerance class is 0, and radial internal clearance is ordinary, and the holder shall be made of punched steel plate.

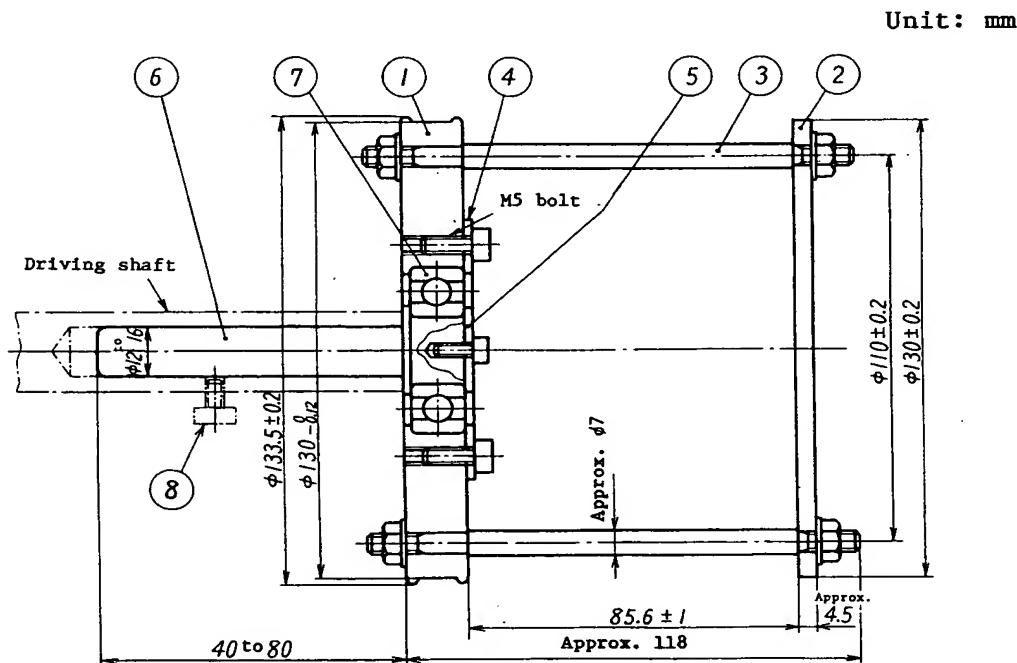
(3) Driving device An example of the driving device is shown in Fig. 35, and shall consist of the motor, the reduction gear, the driving shaft and others. It shall be capable of rotating the driving shaft, provided with spindle of the test jig to it, at a rate of one revolution per min.

Fig. 35. Driving device (An example)



(4) Test jig Jig for the test is shown in Fig. 36 and Fig. 37 in shape and dimensions, and its material shall be stainless steel SUS304 or brass.

Fig. 36. Construction of test jig (An example)

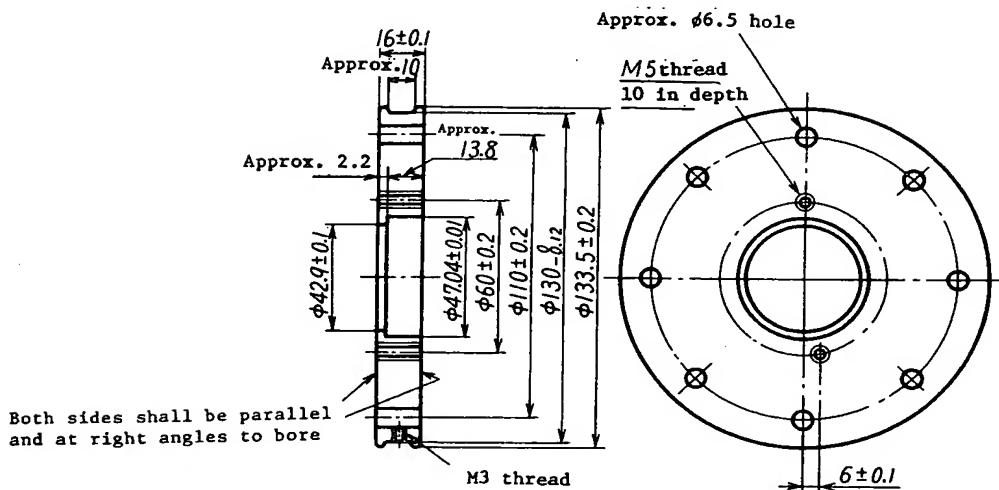


(1) Bearing housing	(5) Inner ring retainer of bearing
(2) Load disk	(6) Spindle
(3) Connecting rod	(7) NP 6204 bearing
(4) Outer ring retainer of bearing	(8) Fixing bolt of spindle

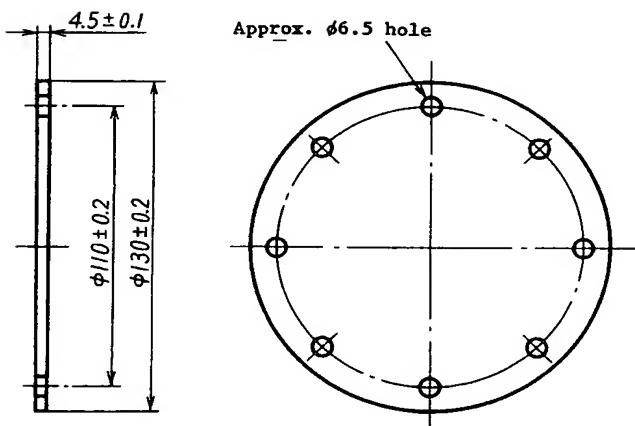
Fig. 37. Parts consisting test jig (An example)

Unit: mm

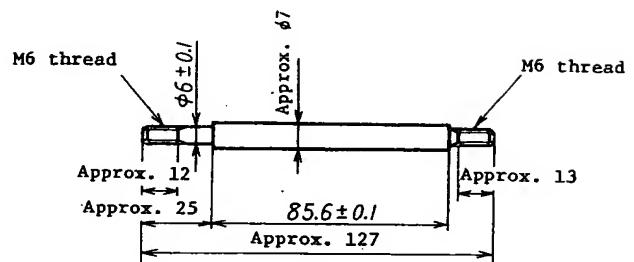
## (1) Bearing housing



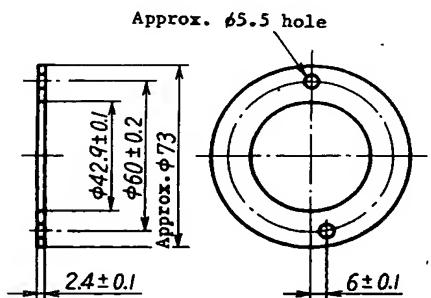
## (2) Load disk



## (3) Connecting rod



## (4) Outer ring retainer of bearing



## (5) Inner ring retainer of bearing

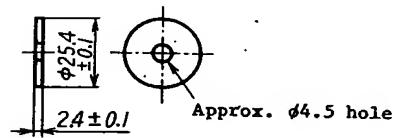
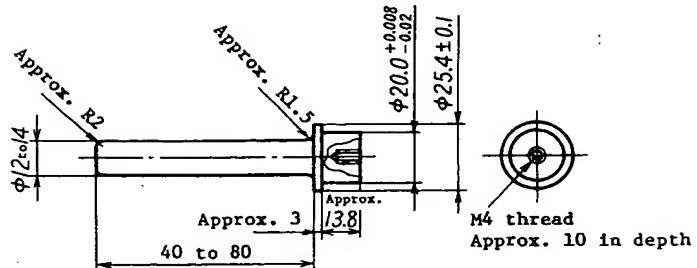


Fig. 37. (continued)

(6) Spindle

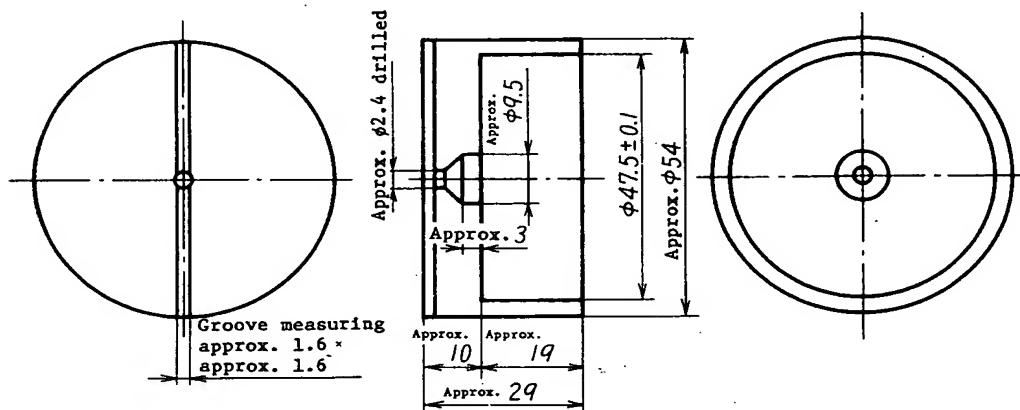


(5) Torque measuring device An instrument capable of measuring the load of 0 N to 39 N{0 kgf to 4.0 kgf}.

(6) Grease cup The grease cup shall be as specified in Fig. 38 in shape and dimensions, and made of brass.

Fig. 38. Grease cup (An example)

Unit: mm



5.14.3 Reagent The solvent shall be petroleum benzine specified in JIS K 8594.

5.14.4 Preparation of test After washing the bearing in accordance with 5.14.5 (1) and drying, when it has been turned in radial and thrust directions with fingers applying slight loads respectively, the bearing shall be able to rotate smoothly. Then apply to bearing several drops of ISO VG 460 specified in JIS K 2238 or of lubricating oil with a viscosity equivalent thereto, and then carry out the rotating torque test at room temperature in accordance with 5.14.5 (9) to measure the average torque and the maximum torque. The average torque shall be less than 0.196 N·cm{20 gf·cm} and the maximum torque be less than 0.245 N·cm{25 gf·cm}. When the measured value of torque is not more than these numerical values, the bearing may be used for the test.

5.14.5 Test procedures The test procedures shall be as follows:

- (1) Clean the test bearing with petroleum benzine, and dry at a temperature not exceeding 100°C, and use it after cooling down to room temperature.
- (2) Mount the dried clean bearing on the spindle [(6) in Fig. 37], and retain the inner ring of the bearing by means of the inner ring retainer and the screw. Fill the grease cup to about 3/4 volume of the cup with the sample grease using a clean steel spatula.
- (3) In order to fill the whole bearing with the sample grease, push the bearing down into the grease while rotating the inner ring and the spindle first in one direction and then in the counter direction slowly. When the bearing reaches the bottom of the cup, take it out, and remove it from the spindle. Turn out the bearing, and refasten it on the spindle. Repack the grease into the grease cup. Again push the bearing into the cup until it reaches the bottom. Remove the spindle and bearing as a unit from the grease cup. Scrape off the excess grease extending on both sides of bearing, and fill holes or clearances left, if any. Take care not to rotate the bearing at any time after packed with the sample grease until commencement of measuring starting torque.
- (4) Mount the bearing packed with sample into the housing, and fasten the outer ring retainer [(4) in Fig. 37] not to let the bearing rotate. Construct the test jig as in Fig. 36.
- (5) Open the low temperature air bath, and slide the spindle of the test jig to the end of the driving shaft.
- (6) Fix the string with screw to the periphery of housing. Connect the string to the needle pointer type spring balance for measurement of torque and rotate<sup>(29)</sup> the spindle until the slack of string is almost removed, then fix the spindle at that position on the driving shaft by means of the bolt.

Note <sup>(29)</sup> At this time, operate so as not to rotate the bearing. The position of the screw on the periphery of housing shall be at least 90° to the vertical.

Remarks: Coat the string with silicone oil to prevent the string from stiffening due to moisture in the air bath at low temperature.

- (7) Close the low temperature air bath, lower the bath temperature to the specified temperature, and maintain this temperature for 2 h<sup>(30)</sup>.

Note <sup>(30)</sup> During this time, do not move the bearing. If it is moved, the test shall be invalidated.

Remarks: Since high humidity in the bath has a possibility to cause condensation of moisture on the bath walls, it is preferable to spread some drying agent, such as activated alumina on a suitable shallow tray and place it on the bottom of the low temperature air bath prior to the test.

(8) Check the string to confirm that it is free of ice, and it is not contacting the bath prior to test.

Remarks: Closing the hole with a split rubber stopper or cotton during the chilling is preferable for keeping the string and its passage free of ice.

Remove the stopper at the time of the measurement of torque.

(9) Start the motor watching the pointer of the needle pointer type spring balance. Record the maximum reading reached<sup>(31)</sup>. Continue the rotation for 10 min, and record the average value of needle pointer type spring balance readings during the last 15 s<sup>(32)</sup>.

Notes <sup>(31)</sup>) The maximum value will occur within a few seconds after starting the rotation.

<sup>(32)</sup>) The temperature of the low temperature air bath shall be maintained at the specified temperature  $\pm 1^{\circ}\text{C}$  during the rotation test.

(10) When the same sample is repetitively tested, clean the bearing in accordance with (1), and pack with a fresh charge of grease in accordance with (2) and (3).

**5.14.6 Calculation and result** Calculate the starting torque ( $\text{N}\cdot\text{cm}\{\text{gf}\cdot\text{cm}\}$ ) and running torque ( $\text{N}\cdot\text{cm}\{\text{gf}\cdot\text{cm}\}$ ) according to the following formula, and round off to significant two figures in accordance with JIS Z 8401 to be taken as the test result.

Starting torque:  $A \times \gamma$

Running torque:  $B \times \gamma$

where,  $A$  : maximum value of needle pointer type spring balance reading just after starting ( $\text{N}\{\text{gf}\}$ )

$B$  : average value of needle pointer type spring balance readings during the last 15 s period for rotation of 10 min ( $\text{N}\{\text{gf}\}$ )

$\gamma$  : torque radius of housing (6.5 cm)

### **5.15 Test method for apparent viscosity**

**5.15.1 Summary of test method** Extrude grease from a cylinder through a capillary by oil hydraulic pressure, and measure the pressure generated in the system at that time. Calculate the apparent viscosity from the pre-determined flow rate, the radius and the length of the capillary, and the measured pressure by the use of Poiseuille's formula defined in 5.15.5. This test method is applicable to the measurement in the temperature range from  $-55^{\circ}\text{C}$  to  $40^{\circ}\text{C}$  and in the apparent viscosity range from 2.5 Pa·s to 10000 Pa·s{25 P to 100000 P} at shear rate  $0.1 \text{ s}^{-1}$  and that of 0.1 Pa·s to 10 Pa·s{1 P to 100 P} at  $15000 \text{ s}^{-1}$ .

5.15.2 Testing apparatus for apparent viscosity The testing apparatus for apparent viscosity shall be composed of the following respective parts (1) to (5). An example of the testing apparatus for apparent viscosity is given in Fig. 39.

Remarks: When test is carried out at other temperature than room temperature, a thermostatic liquid bath or air bath capable of maintaining the temperature of the grease system [Fig. 39 (b)] at the test temperature  $\pm 0.5^{\circ}\text{C}$  shall be used.

Fig. 39. Testing apparatus for apparent viscosity (An example)

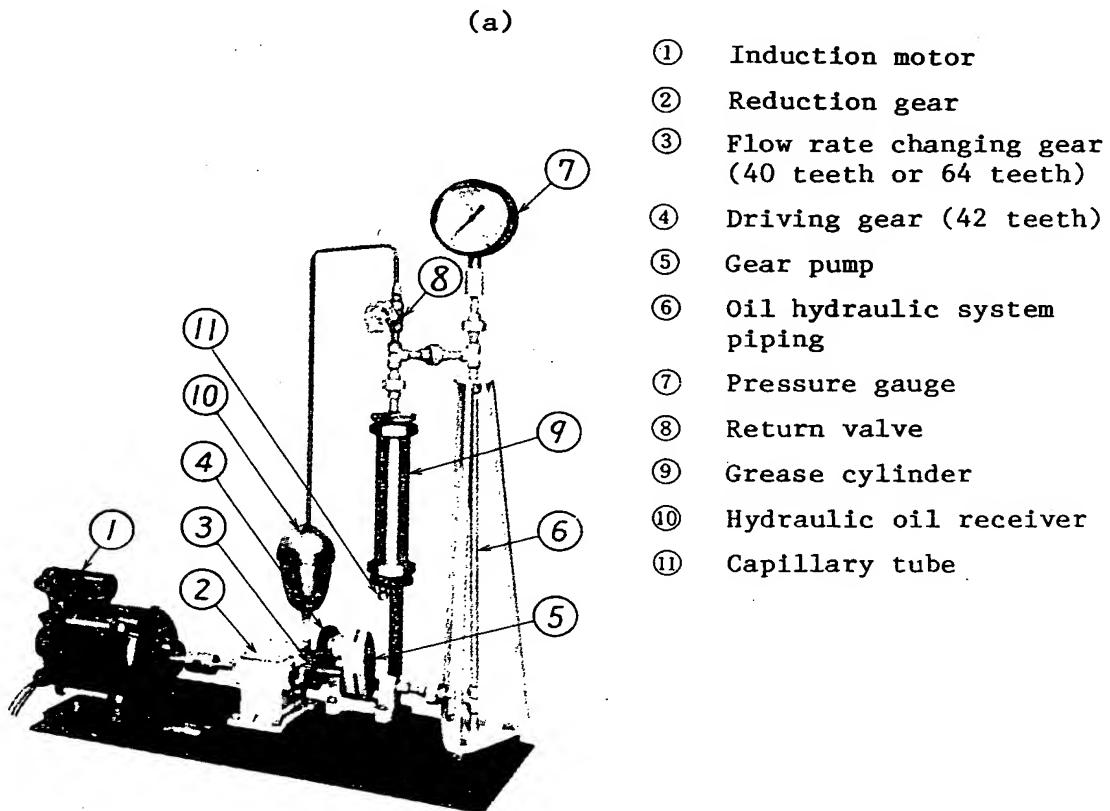
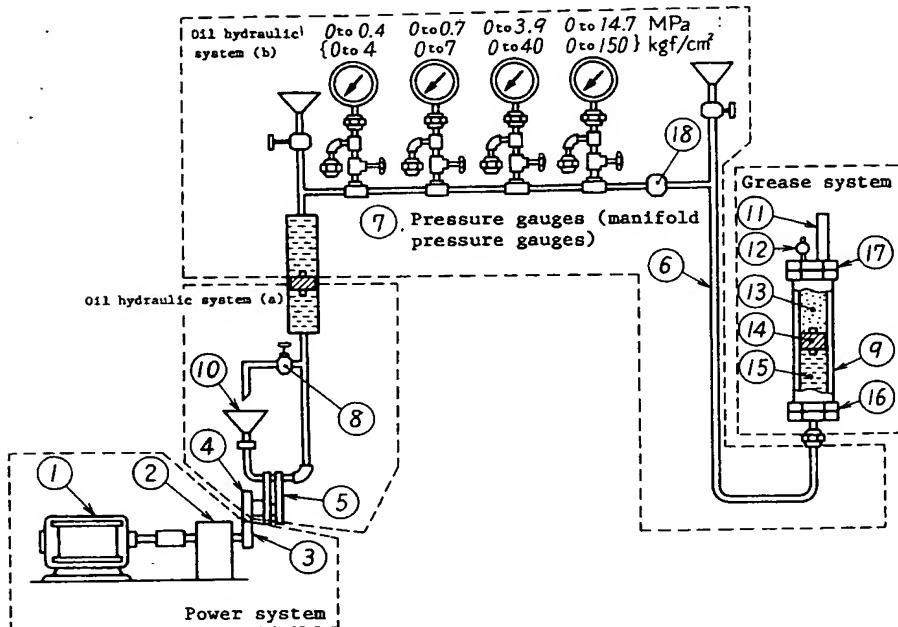


Fig. 39. (continued)

(b)



(12) Temperature sensing element (thermo-couple, thermistor, etc.)	(15) Hydraulic oil
(13) Sample (grease)	(16) End cap A
(14) Piston	(17) End cap B
	(18) Setting position of needle valve

(1) Power system The power system shall consist of about 200 W induction motor and a suitable reduction gear for flow rate changing. Two interchangeable gears, 40 teeth and 64 teeth respectively, shall be used for flow rate changing.

(2) Oil hydraulic system Oil hydraulic system shall consist of gear pump, driving gear of 42 teeth, oil hydraulic system piping and pressure gauges.

(a) Gear pump The pump shall be a constant volume gear pump without pulsations having the delivery pressure of 0 MPa to 27.5 MPa{0 kgf/cm<sup>2</sup> to 280 kgf/cm<sup>2</sup>}, and small variation of delivery volume against pressure fluctuations. The flow rate shall be interchangeable to about 4.8 ml/min and about 7.6 ml/min by the use of 40 teeth gear and 64 teeth gear, respectively.

Remarks: The gear pump shall be equivalent to Type B1 gear pump manufactured by ZENITH, having delivery volume of 0.584 ml/rev. or 1.168 ml/rev., and the hydraulic oil having about 2000 mm<sup>2</sup>/s{cSt} at the temperature of testing room shall be used.

(b) Pressure gauges Pressure gauges shall be as specified in JIS B 7505. For example, gauges having the scale range of 0 MPa to 0.4 MPa{0 kgf/cm<sup>2</sup> to 4 kgf/cm<sup>2</sup>}, 0 MPa to 0.7 MPa{0 kgf/cm<sup>2</sup> to 7 kgf/cm<sup>2</sup>}, 0 MPa to 3.9 MPa{0 kgf/cm<sup>2</sup> to 40 kgf/cm<sup>2</sup>} and 0 MPa to 14.7 MPa{0 kgf/cm<sup>2</sup> to 150 kgf/cm<sup>2</sup>} shall be used.

(c) Oil hydraulic system piping The pipings are to apply hydraulic pressure from the oil hydraulic apparatus to the grease cylinder, and they shall be provided with manifold tubes for pressure gauges and a return valve.

The manifold tubes for pressure gauge may adopt either one gauge mounting shown in Fig. 39 (a) or multiple mounting shown in Fig. 39 (b). When multiple mounting is adopted for fitting of pressure gauges, each tube shall be provided with stop valve.

Pipings of oil hydraulic system shall allow no leakage to a oil pressure of 27.5 MPa{280 kgf/cm<sup>2</sup>}, and shall be arranged so as not to allow air bubbles to stay in the system.

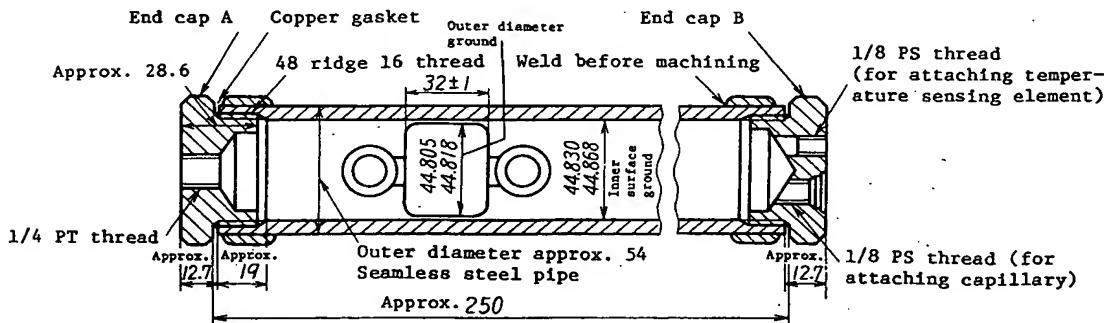
(3) Grease system The system shall consist of grease cylinder part and capillaries.

(a) Grease cylinder part The part is shown in Fig. 40 as an example, and shall withstand a dynamic pressure of 27.5 MPa{280 kgf/cm<sup>2</sup>}. The piston shall be such that which generates no sensible friction when it moves in the cylinder. End cap A [Fig. 39 (b)] shall be capable of being connected to oil hydraulic system, and end cap B [Fig. 39 (b)] shall be capable of being fitted with capillary and temperature sensing element.

(b) Capillary Capillaries are shown in Fig. 41 as an example. Eight capillaries having different inside diameter comprise a set. Diameter of each capillary shall be nearly same as that shown in Fig. 41. The length shall be 40 times the actual diameter.

Fig. 40. Grease cylinder part (An example)

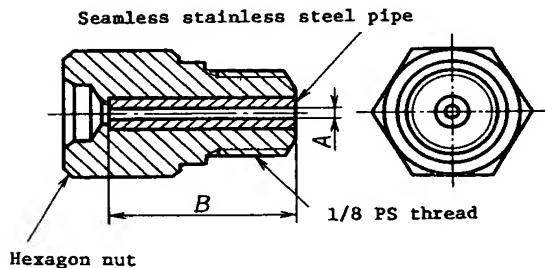
Unit: mm



Remarks: The inner diameter of the cylinder and the outer diameter of the piston shown in the figure indicate the standard dimensions, and alteration is allowed to some degree. However, the clearance between the inner diameter of the cylinder and the outer diameter of the piston shall be 0.012 mm to 0.063 mm. The gasket shall be a copper gasket or a O-ring of synthetic rubber, which shall be so fastened as to produce no leakage under the oil pressure of 14.7 MPa {150 kgf/cm<sup>2</sup>}.

For the fastening, cap nut may be used.

Fig. 41. Capillary (An example)



$$40 \times A(\text{actual diameter}) = B \text{ (length)} \\ \pm 0.002 \text{ cm}$$

No. of capillary	Diameter (approx.) cm
1	0.380
2	0.240
3	0.185
4	0.150
5	0.120
6	0.100
7	0.065
8	0.045

(4) Thermometer the thermometer shall be able to measure the sample temperature in the grease cylinder, and shall be temperature sensing element such as thermocouple or thermistor, capable of being fixed to the end cap B of grease cylinder.

### 5.15.3 Calibration

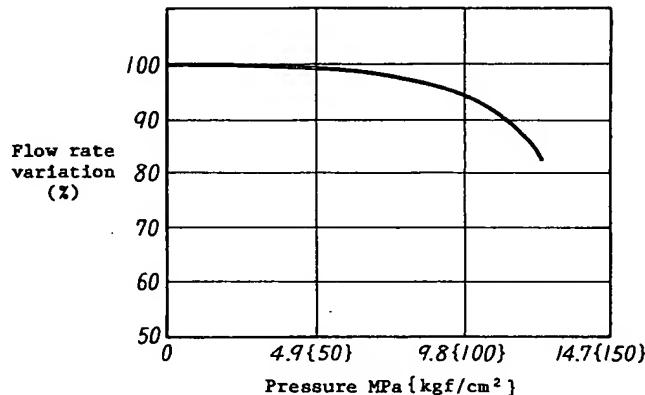
(1) Calibration of flow rate in oil hydraulic system Fill the oil hydraulic system (a)[see Fig. 39 (a)] with hydraulic oil of about 2000 mm<sup>2</sup>/s{cSt} at the temperature of the testing room, then dismount the grease cylinder which has been mounted to oil hydraulic system (b) [see Fig. 39 (b)], and fix the needle valve [Fig. 39 (b) ⑯].

Fill the oil hydraulic system with hydraulic oil having viscosity about 2000 mm<sup>2</sup>/s{cSt} at test temperature, and expel air bubbles.

Maintain the oil hydraulic system (b) shown in Fig. 39 (b) at test temperature, operate the pump under the pressure of 0 MPa{0 kgf/cm<sup>2</sup>}, place quickly a receiving flask for correction of flow rate of oil hydraulic pump under the discharge port, and, at the same time, push the stopwatch. Measure the time required for flowing out of 60 ml of hydraulic oil, and calculate the flow rate (cm<sup>3</sup>/s). Then continue the measurement of the flow rate under the pressure of 2.9 MPa{30 kgf/cm<sup>2</sup>}, 6.9 MPa{70 kgf/cm<sup>2</sup>}, 9.8 MPa{100 kgf/cm<sup>2</sup>}, and 9.8 MPa{100 kgf/cm<sup>2</sup>} or over by adjusting the needle valve. Prepare calibration curve as shown in Fig. 42. This curve is used for the calibration of flow rate when measuring that of sample.

The calibration of oil hydraulic system may be carried out by measuring a flow rate of grease to be tested. Since the wear of the pump changes its flow rate due to abrasion, repeat the calibration at a fixed interval of operation.

Fig. 42. Example of calibration curve for pump flow rate



(2) Calibration of capillary Measure the length of capillary to the nearest 0.001 cm by means of a micrometer. For measuring the inside diameter of a capillary, seal one end of the capillary and erect vertically, fill the capillary with mercury, transfer the mercury into a weighing bottle of known mass, and weigh the mercury to the nearest 1 mg. Calculate the volume by dividing the mass by the density of mercury at the measuring temperature, and calculate the radius of the capillary to the nearest 0.001 cm.

For the calibration of finer diameter of capillary, by means of the procedures specified in 5.15.4, measure the pressure of an oil of known viscosity in place of grease, and calculate the radius of capillary to three places of decimals according to the following formula:

$$R = \left[ \frac{8 L \eta v / t}{10^6 \pi P} \right]^{\frac{1}{4}} = \left[ \frac{8 L \eta v / t}{980.665 \pi P} \right]^{\frac{1}{4}}$$

where,  $R$  : radius of capillary (cm)

$L$  : length of capillary (cm)

$\eta$  : viscosity of used oil at test temperature (Pa·s{P})

$v/t$  : flow rate of pump ( $\text{cm}^3/\text{s}$ )

$P$  : reading of pressure gauge (MPa{kgf/cm²})

#### 5.15.4 Test procedures The test procedures shall be as follows [see Fig. 39 (b)].

(1) Fill the oil hydraulic system (a) with the hydraulic oil of about  $2000 \text{ mm}^2/\text{s}[\text{cSt}]$  in viscosity at test room temperature and the oil hydraulic system (b) with the hydraulic oil of about  $2000 \text{ mm}^2/\text{s}[\text{cSt}]$  in viscosity at the specified test temperature taking care for air bubbles so as not to enter the hydraulic oil.

- (2) Prepare the sample of 0.3 kg or more.
- (3) Fill the grease cylinder with the sample grease with care not to allow air bubbles to enter. Mount the piston into the end cap A side of the grease cylinder, and mount the end cap A, the end cap B and No. 1 capillary.
- (4) Connect the end cap A with the oil hydraulic system (b) while filling the connecting part with hydraulic oil.
- (5) Open the return valve, start the pump to circulate hydraulic oil, and expel air contained in the system. Thereafter, stop the pump and close the return valve.
- (6) Measure the temperature of the sample with the thermocouple, thermistor or the like inserted in the end cap B, and adjust the temperature at the test temperature  $\pm 0.5^{\circ}\text{C}$ (<sup>33</sup>).

Note (<sup>33</sup>) The time required for the sample grease to reach the test temperature is, for example, approximately 2 h for liquid bath and approximately 8 h for air bath, in the case of the test temperature of  $50^{\circ}\text{C}$ .

- (7) When the sample reaches the test temperature, connect the 40 teeth flow rate changing gear, operate the pump, and, when the equilibrium pressure is obtained, record the pressure at that time. Then change the gear to 64 teeth gear, and again obtain the equilibrium pressure. Record the pressure, open the return valve, and release the pressure in the system. Exchange No. 1 capillary for No. 2 capillary, and repeat the procedures above described. Obtain two equilibrium pressures at two flow rates on each capillary, and record the pressures.

5.15.5 Calculation and result The calculation and the result shall be as follows:

- (1) Calculate the apparent viscosity of grease according to the following formula, and obtain to three significant figures:

An example of the calculation is given in Table 22.

$$\eta = F/S$$

where,  $\eta$  : apparent viscosity ( $\text{Pa}\cdot\text{s}$ )  $\{\text{dyn}\cdot\text{s}/\text{cm}^2 = \text{P}\}$

$F$  : shear stress ( $\text{N}/\text{m}^2$ )  $\{\text{dyn}/\text{cm}^2\}$

$S$  : shear rate ( $\text{s}^{-1}$ )

$$\text{so that } \eta = \frac{F}{S} = \frac{10^6 \pi R^4 P}{8 L v/t} = \left\{ \frac{980.665 \pi R^4 P}{8 L v/t} \right\}$$

where,  $P$  : reading of pressure gauge (MPa)  $\{\text{kgf}/\text{cm}^2\}$

$R$  : radius of capillary (cm)

$L$  : length of capillary (cm)

$v/t$  : flow rate ( $\text{cm}^3/\text{s}$ )

Table 22. Example of calculating table for apparent viscosity

Sample: Grease A      Test temp.: 25°C

Capillary	Number of teeth of flow rate changing gear	Reading of pressure gauge $P$ (MPa)	$K = \frac{10^6 \pi R^4}{8 L v/t}$	Apparent viscosity $\eta$ (Pa · s) = $P \times K$	Shear rate <sup>(34)</sup> $S$ (s <sup>-1</sup> ) = $\frac{4v/t}{\pi R^2}$	Shear stress $F$ (N/m <sup>2</sup> ) = $\eta \times S$
1	40	0.175	410	71.8	15	1 080
2	40	0.263	102	26.8	61	1 630
3	40	0.335	52.5	17.6	120	2 110
4	40	0.431	27.8	12.0	230	2 760
5	40	0.657	13.1	8.61	480	4 130
6	40	0.853	8.50	7.25	755	5 470
7	40	1.96	2.03	3.98	3 140	12 500
8	40	3.73	0.679	2.53	9 320	23 600
1	64	0.206	253	52.1	24	1 250
2	64	0.314	62.1	19.5	98	1 910
3	64	0.412	32.9	13.6	195	2 650
4	64	0.569	17.2	9.79	370	3 620
5	64	0.892	8.12	7.24	770	5 570
6	64	1.13	5.31	6.00	1 220	7 320
7	64	2.65	1.26	3.34	5 020	11 800
8	64	4.90	0.426	2.09	14 900	31 100

Note <sup>(34)</sup> Values in this column have been obtained by calculation made in advance.

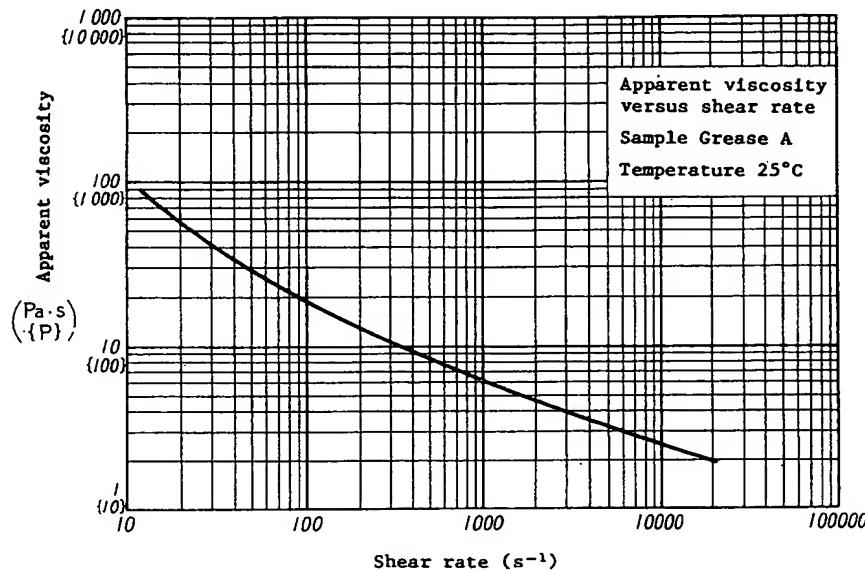
(2) Calculate shear rate according to the following formula.

$$S = \frac{4 v/t}{\pi R^2}$$

Remarks: Sixteen values of shear rate can be obtained by calculation using 8 capillaries and two flow rates.

(3) Prepare the curve of apparent viscosity versus shear rate on a logarithmic paper shown in Fig. 43. From this chart obtain the apparent viscosity at a specified shear rate.

Fig. 43. Example of curve of apparent viscosity versus shear rate



#### 5.15.6 Measuring method for apparent viscosity at low shear rate

(1) Apparatus The apparatus shall be as specified in 5.15.2. However, the capillary shall be of No. 0<sup>(35)</sup>.

Remarks: Since the pressure is low when measuring at the low shear rate, the error shall be minimized as possible by the complete verification of the apparatus and by ensuring the good operating conditions.

Note <sup>(35)</sup> Dimensions of No. 0 capillary

Diameter  $9.525 \pm 0.025$  mm

Length  $381.000 \pm 0.025$  mm

(2) Test procedures The procedures shall be as specified in 5.15.4.

Remarks: When measuring at the shear rate of  $1\text{ s}^{-1}$  or less, it is preferable to use a variable flow rate pump.

(3) Calculation and result The calculation and the result shall be as specified in 5.15.5.

#### 5.16 Test method for load carrying capacity by Timken machine

##### 5.16.1 Summary of test method

Summary of the test method shall be as follows:

(1) This test method specifies the items necessary for testing load carrying capacity of grease by the Timken's method specified in 6. of JIS K 2519\*, and the definitions of terms, outlines of tester and the test procedure shall be in accordance with JIS K 2519.

\* Translator's note: JIS K 2519 referred in this Standard is of 1987 edition.

(2) Obtain OK value and score value from the condition of abrasion trace found on the test block after 10 min driving of the specified tester under fixed load while supplying the grease at constant feeding rate.

**5.16.2 Apparatus and devices** The Timken's extreme pressure tester, test cup, test block, automatic loading device, magnifying glass or microscope, and watch each specified in 6.2 of JIS K 2519 shall be used, and, in addition to them, the grease feeding device described below shall be used.

The grease feeding device shall be as shown in Fig. 44. It shall have a container of a capacity enough to feed the sample to be used for one test, and shall be provided with a suitable piston mechanism capable of feeding grease at a rate of  $45 \pm 9$  g/min.

This feeding device shall be fixed to Timken's tester, as shown in Fig. 45, from which the upper sample container has been detached previously.

**5.16.3 Cleaning solvent** As described in 6.3 of JIS K 2519.

**5.16.4 Preparation of test** Preparation of the test shall be as follows:

(1) Detach the upper sample container of Timken's tester, and fix the grease feeding device to it. Since the pump is not used for grease test, disconnect the joint of pump from the revolving shaft for prevention of abrasion due to no fluid running.

**Remarks:** In order to avoid the abrasion of pump, it is preferable to install previously a piping system having 3-way cock and a grease reservoir to a part of the grease circulating system of Timken's tester.

(2) Thereafter, follow the procedures specified in 6.4 of JIS K 2519.

**5.16.5 Test procedure** The test procedure shall be as follows:

(1) Use the grease at  $24 \pm 6$  °C. Fill the grease feeding device with the sample grease with care not to allow air bubbles to enter. Coat the sample thinly over the test cup and the block.

(2) Operate the grease feeding device, and feed the grease to the test surface at a rate of  $45 \pm 9$  g/min.

Then drive the revolving shaft of the tester to carry out a running-in for about 30 s.

(3) Thereafter, follow the procedures specified in 6.5.1 of JIS K 2519 to obtain OK value and score value.

**5.16.6 Result** Obtain OK value and score value in accordance with 6.6 of JIS K 2519, and when necessary, calculate the contact pressure between the test cup and the test block at the time when OK value is obtained.

Fig. 44. Grease feeding device (An example)

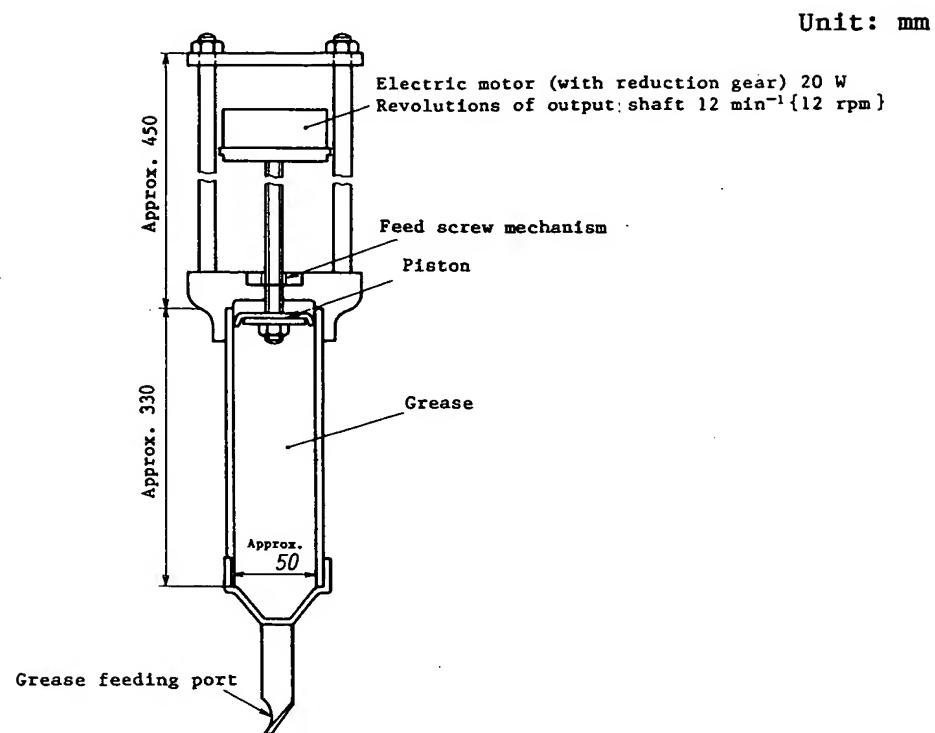
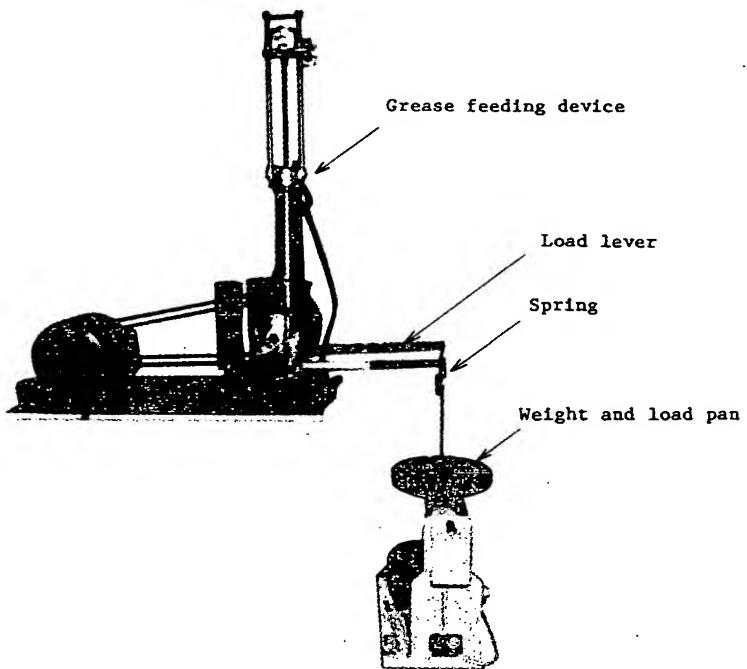


Fig. 45. Assembly of grease feeding device (An example)



**Informative reference:** The weights of Timken's extreme pressure tester on the market are, in most cases, made in pounds unit. For the convenience of the users, the relation between lb and kg given for reference in Informative reference Table 1.

Informative reference Table 1. Relation between lb and kg

lb	kg
60	27.2
55	25.0
50	22.7
45	20.4
40	18.1
35	15.9
30	13.6
27	12.2
24	10.9
21	9.53
18	8.16
15	6.80
12	5.44
9	4.08
6	2.72
3	1.36

### 5.17 Method for humidity cabinet test

5.17.1 Summary of test method Hang a steel plate coated with a sample in a humidity cabinet, which keeps the temperature of 49°C, and relative humidity of 95 % or over. After specified time has elapsed, examine the degree of rust gathering.

5.17.2 Apparatus and device The apparatus and device shall consist of the following (1) to (3).

- (1) Test piece As specified in 5.3.1 of JIS K 2246.
- (2) Humidity cabinet test apparatus As specified in 5.34.2 (1) of JIS K 2246.
- (3) Measuring plate for degree of rust gathering As specified in 5.4.2 of JIS K 2246.

5.17.3 Reagent Solvent shall be as specified in JIS K 8594.

5.17.4 Preparation of test piece As specified in 5.3.1 and 5.3.2 of JIS K 2246.

5.17.5 Preparation of coated test piece Coat the sample uniformly all over the surface of the test piece prepared in accordance with 5.17.4 so that the coated amount on the one surface shall be  $0.30 \pm 0.05$  g in a suitable way<sup>(36)</sup>.

Note<sup>(36)</sup> Grease to be used for the test shall be such that foams of which have been previously removed thoroughly. It is preferable that after putting an appropriate amount of the grease on one surface of the test piece, the grease is coated little by little like rubbing while being pressed strongly onto the test piece with a spatula.

5.17.6 Test procedures Test procedures shall be as follows:

- (1) The test shall be made following the procedures specified in 5.34.3 (1) and (2) of JIS K 2246.
- (2) After specified time elapsed, take out the test piece, select the test surface facing to the rotating direction of the frame on which the test piece is mounted as the surface to be measured, remove the sample grease with a clean cloth, further wipe off the grease with petroleum benzine, and measure the degree of rust gathering (%) in accordance with 5.4.3 of JIS K 2246.

5.17.7 Result Take the averaged value of the measurements made for three test pieces having been coated with the same sample as the average degree of rust gathering, classify into 5 classes, 0 %, 1 % to 10 %, 11 % to 25 %, 26 % to 50 % and 51 % to 100 %, and denote them as Classes A, B, C, D and E in the order to express (see Table 11 of JIS K 2246).

5.18 Test method for water content Follow the method specified in 4. of JIS K 2275.

5.19 Test method for kinematic viscosity Follow the method specified in JIS K 2283.

5.20 Test method for flash point Follow the method specified in JIS K 2265.

5.21 Test method for load carrying capacity by four-ball machine Follow the method specified in 5. of JIS K 2519.

6. Designation of products Designation of products shall be made by the classification (class according to use, grade and number of cone penetration).

Example: Grease for general purpose, Class 1, Grade 2

7. Marking The following particulars shall be marked on a conspicuous place of the container in an indelible way.

- (1) Classification (class according to use, grade and number of cone penetration)

Example: Grease for general purpose, Class 1, No. 2

- (2) Net mass
- (3) Manufacturer's name or abbreviation
- (4) Year and month of manufacture or its abbreviation
- (5) Lot number

Attached Table 1. Cited standards

JIS B 1521	Rolling bearings - Deep groove ball bearings
JIS B 7410	Liquid-in-glass thermometers for testing of petroleum products
JIS B 7505	Bourdon tube pressure gauges
JIS G 3459	Stainless steel pipes
JIS G 4051	Carbon steels for machine structural use
JIS G 4105	Chromium molybdenum steels
JIS G 4303	Stainless steel bars
JIS G 4305	Cold rolled stainless steel plates, sheets and strip
JIS H 3100	Copper and copper alloy sheets, plates and strips
JIS H 3250	Copper and copper alloy rods and bars
JIS H 5101	Brass castings
JIS K 1101	Oxygen
JIS K 2238	Machine oils
JIS K 2246	Rust preventive oils
JIS K 2251	Crude petroleum and petroleum products - Sampling
JIS K 2265	Testing methods for flash point of crude oil and petroleum products
JIS K 2275	Testing methods for water content of crude oil and petroleum products
JIS K 2283	Crude petroleum and petroleum products - Determination of kinematic viscosity and calculation of viscosity index from kinematic viscosity
JIS K 2513	Petroleum products - Corrosiveness to copper - Copper strip test
JIS K 2519	Testing methods for load carrying capacity of lubricating oil

Attached Table 1. (continued)

JIS K 2839	Glasswares for testing apparatus of petroleum products
JIS K 6323	Classical V belts for power transmission
JIS K 8034	Acetone
JIS K 8594	Petroleum benzine
JIS R 6111	Artificial abrasives
JIS R 6251	Abrasive cloths
JIS R 6252	Abrasive papers
JIS T 3201	Glass syringes
JIS Z 8401	Rules for rounding off of numerical values
JIS Z 8402	General rules for permissible tolerance of chemical analyses and physical tests
JIS Z 8801	Test sieves

Informative reference 1  
Test method for quarter and half cone penetrations

This Informative reference describes the method for measuring the un-worked penetration and worked penetration of grease when it is small in amount, but does not constitute a part of the Standard.

1. Scope This Informative reference 1 specifies the measuring method for unworked penetration and worked penetration of the sample when it is small in amount.

Remarks 1. Unless the grease is of 0 to 4 in cone penetration number, the sample shall not be furnished to this test.

2. This method applies only when the test method for cone penetration specified in 5.3 of the text of this Standard is inapplicable because of too small amount of the sample.

2. Summary of the test method Allow a quarter cone or a half cone to penetrate in a sample at the temperature of 25°C for 5 s, and thereby measure the unworked penetration and the worked penetration.

3. Testing apparatus for quarter and half cone penetration The apparatus shall be as shown in Informative reference Fig. 1 to Fig. 5, and consist of the parts described in following (1) to (7).

(1) Penetrometer The penetrometer shall be as shown in Informative reference 1 Fig. 1 as an example, equipped with a device capable of measuring the depth to the nearest 0.1 mm when allowing the specified cone (quarter or half cone) to penetrate in the sample perpendicularly, and shall consist of dropping device with retainer, dial gauge, sample table, supporting table with level adjusting screw, and others.

Furthermore, it shall be equipped with a fine vertical adjusting mechanism capable of minutely adjusting the distance between the surface of the sample placed on the table and the tip of the cone attached to the holder at the time of test.

(a) Dropping device A device having such construction that only for the retainer shown in Informative reference 1 Fig. 1 acts, it allows the cone to penetrate in a sample perpendicularly together with the holder. It shall be so constructed as to have extremely little frictional resistance to dropping, and shall have a 20 mm or more moving distance of falling.

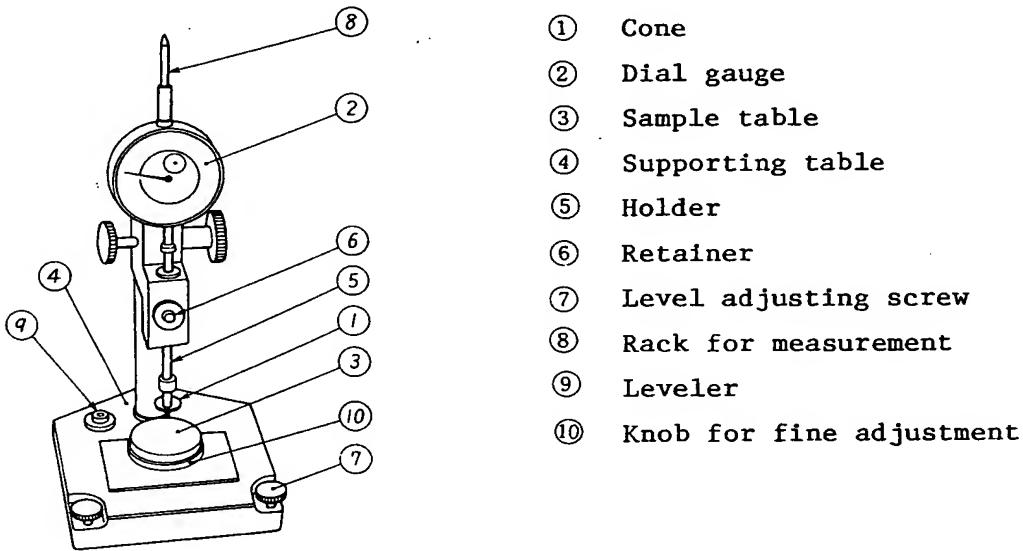
Remarks: A device having such a mechanism that a self-timer or the like activates the retainer automatically may be used.

(b) Holder The holder shall be as shown in Informative reference 1 Fig. 1 and to hold the cone. The holder for quarter cone shall be  $9.38 \pm 0.025$  g in gross mass including the attached cone, and the mass of the half cone holder shall be  $15.00 \pm 0.025$  g.

(c) Dial gauge The dial gauge shall be as shown in Informative reference 1 Fig. 1, and shall have such mechanism that a moving distance is indicated on the scale when the rack for measurement is pressed down and that the moving distance within 20 mm can be read to the nearest 0.1 mm.

(d) Supporting table The supporting table shall be as shown in Informative reference 1 Fig. 1, and consist of the cone dropping device, a supporting shaft to hold a dial gauge, a sample table, and a base equipped with level adjusting screw.

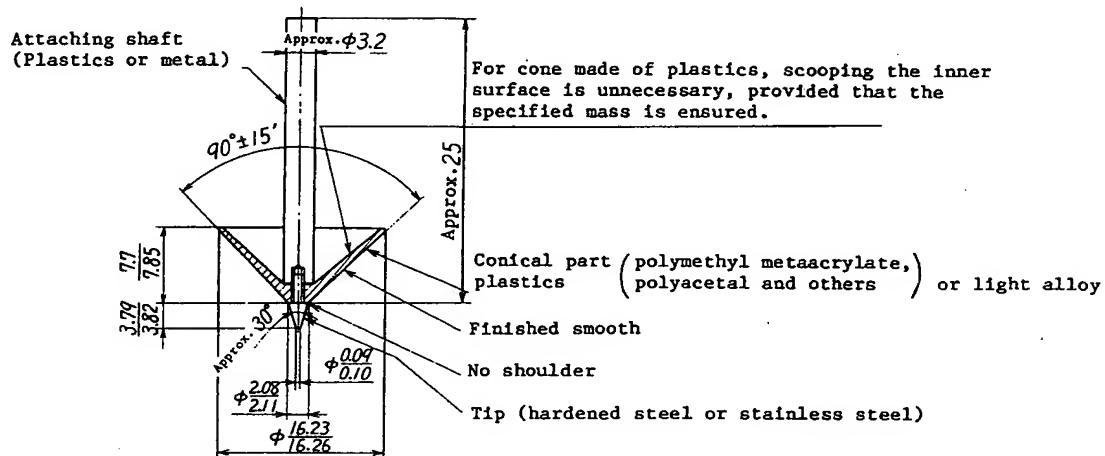
Informative reference 1 Fig. 1. Construction of penetrometer  
(An example)



(2) Quarter cone The cone shall be as shown in Informative reference 1 Fig. 2 as an example, and it shall be finished smooth on the outer surface. It shall be  $9.38 \pm 0.025$  g in gross mass including the holder.

## Informative reference 1 Fig. 2. Quarter cone (An example)

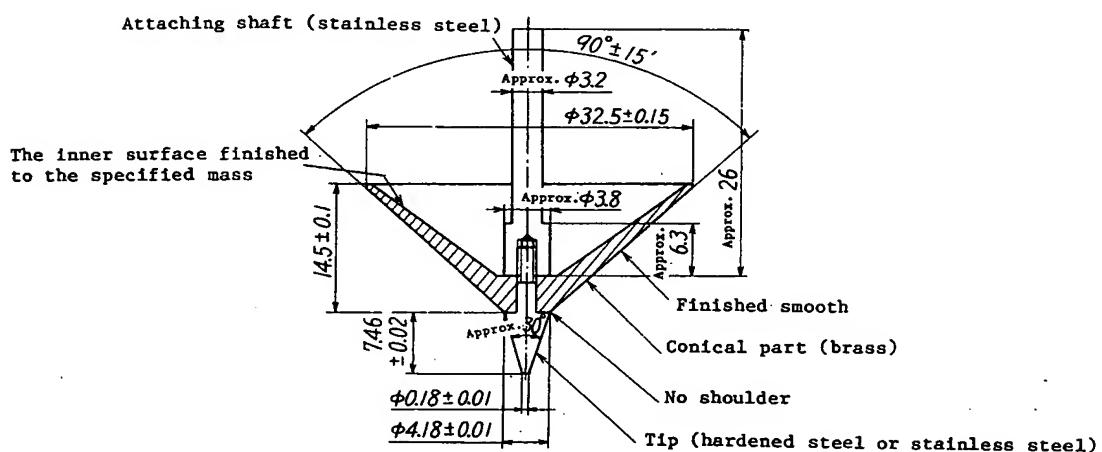
Unit: mm

Mass Gross mass of cone together with holder  $9.38 \pm 0.025$  g

(3) Half cone The cone shall be as shown in Informative reference 1 Fig. 3 as an example, and its outer surface shall be finished smooth. It shall be  $22.5 \pm 0.025$  g in mass.

## Informative reference 1 Fig. 3. Half cone

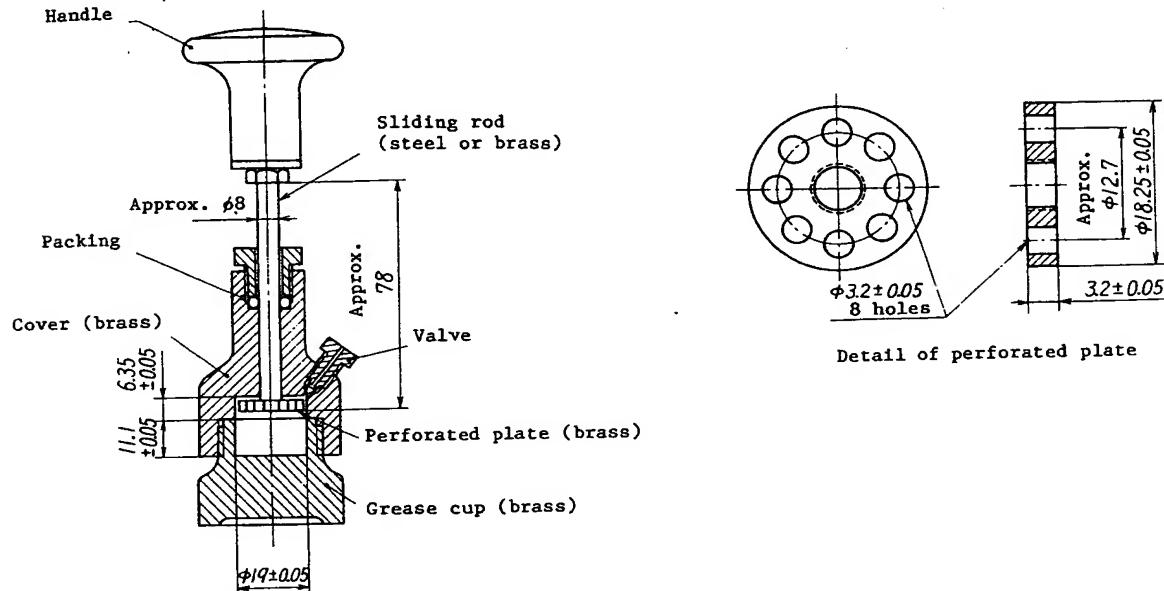
Unit: mm

Mass  $22.5 \pm 0.025$  g

(4) Quarter worker The worker shall be as shown in Informative reference 1 Fig. 4 as an example, and capable of moving up and down the perforated plate attached to the top of the sliding rod.

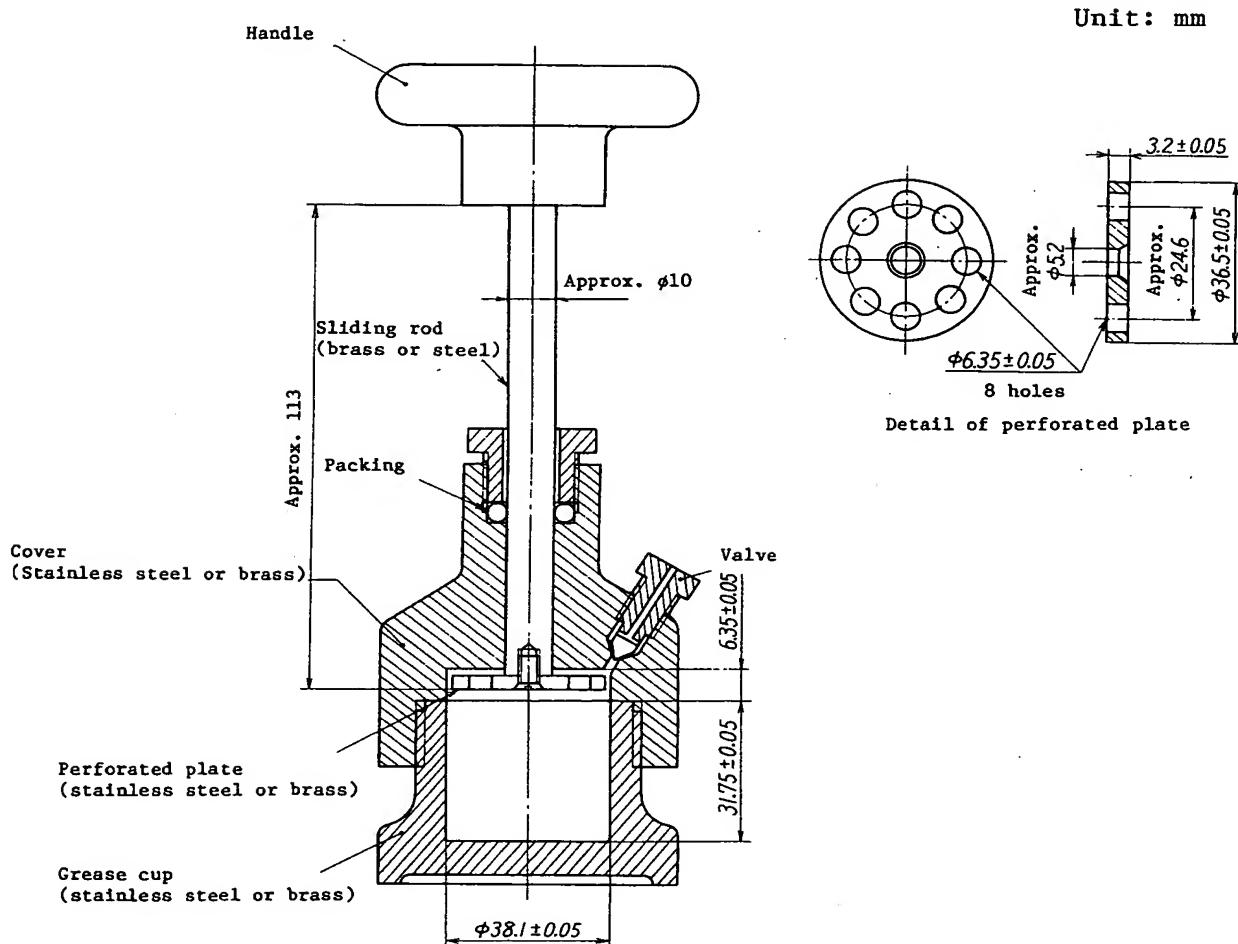
Informative reference 1 Fig. 4. Quarter worker (An example)

Unit: mm



(5) Half worker The worker shall be as shown in Informative reference 1 Fig. 5 as an example, and capable of moving up and down the perforated plate attached to the top of the sliding rod.

Informative reference 1 Fig. 5. Half worker (An example)



- (6) Spatula The spatula shall be made of stainless steel in about 13 mm width and suitable length, and shall have a top of square shape and a grip.
- (7) Thermostatic water bath A suitable water bath capable of keeping the bath temperature at  $25 \pm 0.5^\circ\text{C}$ , and shall provide a suitable shelf which facilitates for immersing the worker in the bath liquid.

#### 4. Measuring method of worked penetration

- (1) Preparation of sample The preparation of the sample shall be made as described below.
  - (a) Prepare an amount of the sample enough to fill the cup of the worker, pack the sample in the clean cup of the worker with a spatula with care not to allow air to enter, and mound it into mountain shape so that the central part is about 5 mm higher than the rim of the cup. Then open the valve of cover, assemble the worker, depress the perforated plate to the bottom of the cup, close the valve, immerse the worker<sup>(2)</sup> in the thermostatic water bath<sup>(1)</sup> kept at  $25^\circ\text{C}$ , and leave it still until the temperature of the sample reaches  $25 \pm 0.5^\circ\text{C}$ <sup>(3)</sup>.

Notes <sup>(1)</sup> Instead of using thermostatic water bath, the thermostatic room or air bath may be used, but in those cases, leaving time shall be taken long enough.

(<sup>2</sup>) When immerse the worker into the thermostatic water bath, it shall be done by using a water-tight-worker or by wrapping the worker with polyethylene bag in order to prevent the entrance of water to the worker.

(<sup>3</sup>) About 2 h will be needed to reach the sample at 25°C.

When the sample temperature is 17°C or under or 33°C or more before immersing into the thermostatic water bath, the leaving time in thermostatic water bath needs to take a long time enough.

(b) Then take out the worker from the thermostatic water bath, wipe off water adhering to its outer surface, close valve and work the sample with 60 double strokes per min by the perforated plate, and return the plate to its top position. Open the valve, remove the cover and perforated plate and return as much of the sample adhering to it as may readily be removed to the cup. Because the worked penetration may change significantly on standing, proceed immediately in accordance with following procedures (c) and (2).

(c) For making the worked sample into a uniform structure, beat the cup sharply on a suitable workbench and pack the sample to fill the clearance caused by working the sample. Scrape off most of the sample with spatula and take out the sample in bottom of the cup onto the surface<sup>(4)</sup>. Then beat the cup strongly on the workbench to remove the air bubble. Extruded sample shall be packed into the cup with a spatula. Flatten the mounded sample<sup>(5)</sup> on the rim of the cup by moving the blade of the spatula, held inclined toward the direction of motion by an angle of about 45°, along the rim of the cup as shown in Fig. 8 in the text of this Standard. From that time on, care shall be taken not to touch the surface of sample with the spatula or the like before measurement.

Notes <sup>(4)</sup> Care shall be paid not to work sample beyond necessity.

<sup>(5)</sup> Retain the excess sample removed from the cup for subsequent test to fill the cup.

(2) Measurement of penetration and result Measure the penetration in accordance with 5.3.3 (2) in the text, and obtain the worked penetration to digit of integer by rounding off the average value of three test results. The fact that either quarter or half cone has been used shall be appended to the test result.

##### 5. Measuring method for unworked penetration

(1) Preparation of sample The preparation of the sample shall be made as described below.

- (a) When the unworked penetration is 47 or less in the case of quarter cone, and 97 or less in the case of half cone, prepare an amount of the sample enough to fill the cup of the worker.
- (b) When the unworked penetration exceeds 47 in the case of quarter cone or exceeds 97 in the case of half cone, prepare an amount of the sample enough to fill three cups of the worker.
- (c) Then proceed in accordance with (1)(b) to (e) of 5.3.4 in the text.

(2) Measurement of penetration

- (a) When the unworked penetration is 47 or less in the case of quarter cone, or 97 or less in the case of half cone, measure the penetration by the same cup in accordance with (2)(a) and (b) in 5.3.3 of the text at the different three positions shown in Fig. 9 of the text.
- (b) When the unworked penetration exceeds 47 in the case of quarter cone, or exceeds 97 in the case of half cone, measure the penetration in accordance with (2)(a) and (b) in 5.3.3 of the text at the center of sample surface.
- (c) After finishing the measurement, take out the sample from the cup, refill the cup with another prepared sample in 5.(1)(b), measure the penetration in accordance with (1)(d) and (e) in 5.3.4 of the text and (2)(a) and (b) in 5.3.3 of the text.

(3) Result Result of unworked penetration from an average of three tests made for the same sample obtained in (2) shall be reported to digit of integer as the rounding off value. The fact that either quarter cone or half cone has been used shall be appended to the test result.

6. Precision The precision shall be as follows:

(1) Repeatability The tolerance on the difference in test results between two tests successively made for the same sample by the same person with the same apparatus in the same testing room in short time shall be given as follows.

	In the case of quarter cone	In the case of half cone
Unworked penetration	3	5
Worked penetration	3	3

(2) Reproducibility The tolerance on the difference in test results between two single tests made for the same sample by different persons with different apparatus in different testing rooms shall be given as follows.

	In the case of quarter cone	In the case of half cone
Unworked penetration	10	13
Worked penetration	7	10

Remarks: When the test result exceeds the tolerance, the result shall be treated in accordance with JIS Z 8402.

7. Conversion of penetration The conversion to the penetration specified in 5.3 in the text can be calculated according to the following formulae:

(1) Conversion from quarter cone penetration

$$P_0 = 3.75 P_4 + 24$$

where,  $P_0$  : penetration to be obtained

$P_4$  : penetration of quarter cone

(2) Conversion from half cone penetration

$$P_0 = 2.00 P_2 + 5$$

where,  $P_0$  : penetration to be obtained

$P_2$  : penetration of half cone

## Informative reference 2

## Method for determination of free acid, free alkali and insoluble carbonate

This Informative reference 2 describes the testing method for calculating free acid, free alkali, and insoluble carbonate, but does not constitute a part of the Standard.

1. Summary of the test method Disperse a sample in solvent, add ethyl alcohol, and drop phenolphthalein solution (10 g/l) as indicator. In the case of acidic indication, calculate the free acid by titration with 0.5 mol/l alcoholic solution of potassium hydroxide. In the case of alkaline indication, add a known amount of 0.5 mol/l hydrochloric acid, boil, neutralize free alkali, back-titrate excess acid with alcoholic solution of potassium hydroxide, and calculate free alkali.

In the case of determination of free alkali, when insoluble carbonate is detected by effervescence, after adding the excess of 0.5 mol/l hydrochloric acid, back-titrate with 0.5 mol/l alcoholic solution of potassium hydroxide, and calculate the insoluble carbonate.

Remarks: This method is inapplicable to the grease which contains weak basic soap, such as lead, zinc, aluminum and others, or the grease containing additives which react on potassium hydroxide and hydrochloric acid.

2. Reagents

- (1) Solvent As specified in JIS K 8594.
- (2) Ethyl alcohol As specified in JIS K 8102 (neutralized).
- (3) Solution of phenolphthalein Prepare as follows: Dissolve 1.0 g of phenolphthalein specified in JIS K 8799 in 50 ml of ethyl alcohol, and add 50 ml of water.
- (4) 0.5 mol/l alcoholic solution of potassium hydroxide<sup>(1)</sup> Prepare as follows: Dissolve about 29 g of potassium hydroxide specified in JIS K 8574 in 500 ml of ethyl alcohol, leave quietly in dark place, filter the supernatant solution in a suitable way, add ethyl alcohol to make up the volume to 1000 ml, and conduct a precise standardization.

Note <sup>(1)</sup> 0.2 mol/l or 0.1 mol/l alcoholic solution of potassium hydroxide may be used.

- (5) 0.5 mol/l hydrochloric acid Dilute about 50 ml of hydrochloric acid specified in JIS K 8180 with water to make up the volume to 1000 ml, and conduct the standardization with 0.5 mol/l alcoholic solution of potassium hydroxide.

3. Apparatus Burettes specified in JIS R 3505 having the capacity of 50 ml (min. graduation 0.1 ml) and that of 10 ml (min. graduation 0.05 ml).

4. Test procedure The test procedure shall be as follows:

- (1) Preparation of sample Weigh accurately about 10 g of sample in a suitable beaker of 100 ml or over to the nearest 0.1 g, add 75 ml of solvent dividing in several portions, disperse the sample thoroughly in the solvent. Then transfer to a 250 ml Erlenmeyer flask, wash out completely the content of the beaker in to the Erlenmeyer flask with 50 ml of ethyl alcohol, add about 1 ml of phenolphthalein solution, shake vigorously to mix at an appropriate interval for 10 min.
- (2) Determination of free acid When the alcoholic layer of (1) does not colour, immediately titrate with 0.5 mol/l alcoholic solution of potassium hydroxide until the faint pink colour continues for 1 min.
- (3) Determination of free alkali When the alcoholic layer of (1) colours, add 50 ml or, when necessary, excess of 0.5 mol/l hydrochloric acid, and decompose thoroughly to make acidic<sup>(2)</sup>. Attach a reflux condenser to the flask, put on a hot plate, boil the content for about 10 min to expel carbon dioxide.

Then detach the reflux condenser, and immediately titrate with 0.5 mol/l alcoholic solution of potassium hydroxide in accordance with (2) as appropriate.

Note <sup>(2)</sup> When the content contains carbonate, vigorous effervescence appears by the addition of hydrochloric acid. In such a case, not clause (3) but clause (4) applies.

- (4) Determination of insoluble carbonate When carbonate is observed, add 0.5 mol/l hydrochloric acid until the content shows slightly acidic, attach a reflux condenser to the Erlenmeyer flask, place on the hot plate, and boil the content for 10 min to expel carbon dioxide. Then detach the condenser, and immediately titrate with 0.5 mol/l alcoholic solution of potassium hydroxide in accordance with (2) as appropriate.

5. Blank test Carry out blank test following the procedures of 4.(2) and (3) respectively. The procedure of 4.(4) does not apply to the blank test.

6. Calculation and result Calculation and result shall be made as follows:

- (1) Calculate free acid as oleic acid and obtain to one place of decimal according to the following formula:

$$A = \frac{28.2 \times (v - v') \times n}{W}$$

where, A : free acid (%)

v : volume of 0.5 mol/l alcoholic solution of potassium hydroxide consumed in titration of sample (ml)

v' : volume of 0.5 mol/l alcoholic solution of potassium hydroxide consumed in blank test (ml)

n : concentration of 0.5 mol/l alcoholic solution of potassium hydroxide (mol/l)

W : mass of sample (g)

(2) Calculate free alkali as hydroxide of principal metal composing a soap and obtain to two places of decimals according to the following formula:

$$B = \frac{E \times (v' - v) \times n}{10 \times W}$$

where,  $B$  : free alkali (%)

$E$  : equivalent mass of hydroxide of principal metal (g)

$v$  : volume of 0.5 mol/l alcoholic solution of potassium hydroxide consumed in titration of sample (ml)

$v'$  : volume of 0.5 mol/l alcoholic solution of potassium hydroxide consumed in blank test (ml)

$n$  : concentration of 0.5 mol/l alcoholic solution of potassium hydroxide (mol/l)

$W$  : mass of sample (g)

(3) Calculate insoluble carbonate as calcium carbonate and obtain to one place of decimal according to the following formula:

$$C = \frac{5 \times (V \times N - v \times n)}{W}$$

where,  $C$  : insoluble carbonate (%)

$V$  : volume of 0.5 mol/l hydrochloric acid added to sample (ml)

$N$  : concentration of 0.5 mol/l hydrochloric acid

$v$  : volume of 0.5 mol/l alcoholic solution of potassium hydroxide consumed for titration of sample (ml)

$n$  : concentration of 0.5 mol/l alcoholic solution of potassium hydroxide (mol/l)

$W$  : mass of sample (g)

Informative reference 3. Test method for evaporation loss

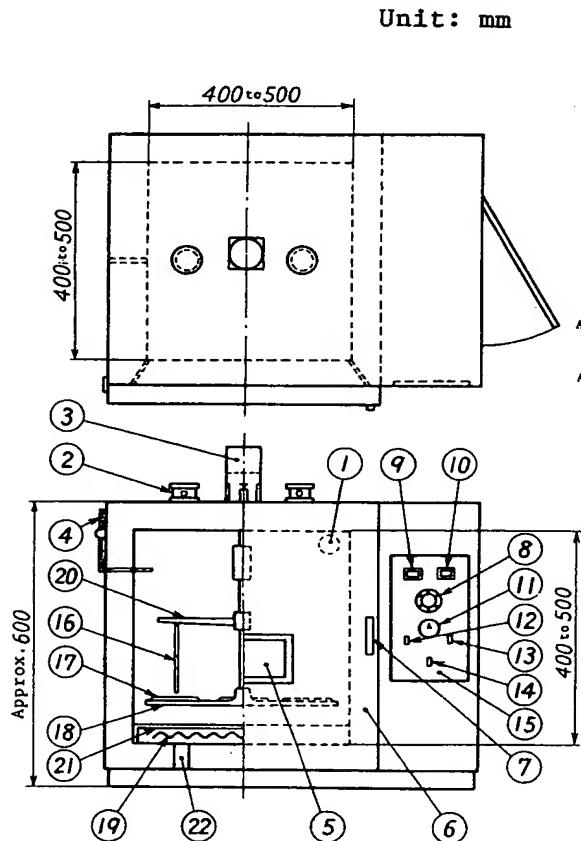
This Informative reference 3 describes the test method for the evaporation loss of grease, and does not constitute a part of the Standard.

1. Summary of test method Heat a sample for 8 h in a thermostatic air bath kept at 105°C, equipped with a rotating disk which turns at a rate of 5 to 6 revolutions per min. Calculate the evaporation loss percentage from the loss in mass of the sample.

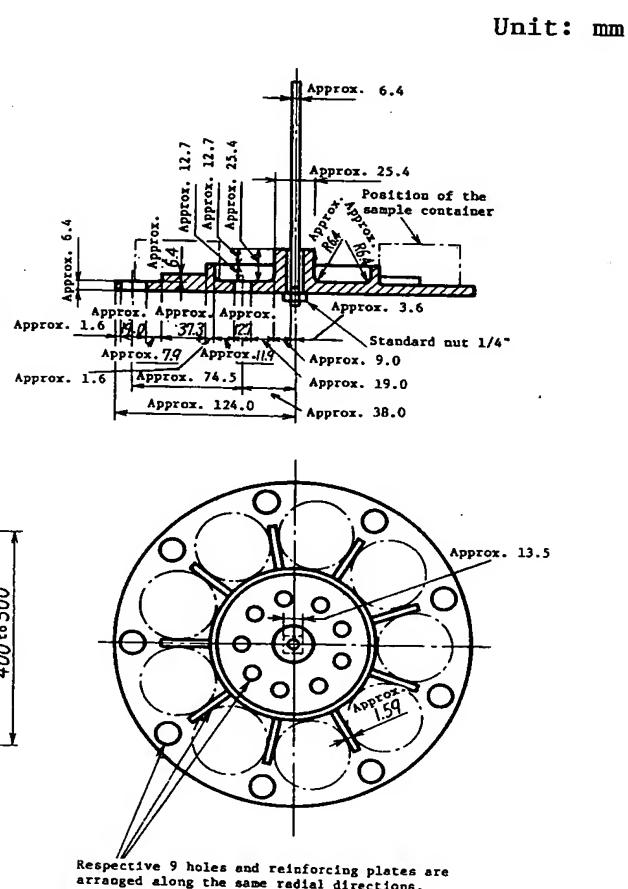
2. Apparatus and device The apparatus and device shall consist of the following items (1) to (3).

- (1) Sample container As specified in Fig. 153 of JIS K 2839.
- (2) Thermostatic air bath An electric heating thermostatic air bath as shown in Informative reference 3 Fig. 1 (a) of double wall, square type, the internal dimensions of which shall be 290 mm or over in height excluding the heating part, and 300 mm or over both in width and depth. It shall be equipped with a swinging door, and on the wall a square double glass window of one side length approximately 100 mm to observe the temperature and the inner state. The thermostatic air bath shall be equipped with one or more air inlets and at least one hot air and vapour outlet to facilitate the suitable ventilation. For circulating air around the heating coil, the air inlets shall be located at the symmetrical positions of the bottom part or the lower part of side wall, and the total sectional area shall be 130 mm<sup>2</sup> or over. Outlets of hot air and vapour shall be located at the symmetrical positions of the top or the side wall in the vicinity of top, and the total sectional areas shall be 130 mm<sup>2</sup> to 1290 mm<sup>2</sup>. At the inner center of thermostatic air bath, an aluminum rotating disk of about 250 mm diameter made to the shape shown in Informative reference 3 Fig. 1 (b), capable of rotating at a rate of 5 to 6 revolutions per min by an electric motor, shall be suspended.
- (3) Thermometer The thermometer shall have about 150 mm in total length, the graduation range of 100°C to 110°C, and the scale interval of 0.5°C.

## Informative reference 3 Fig. 1.

(a) Thermostatic air bath  
(An example)

(b) Rotating disk made of aluminum (An example)



① Inside lamp of air bath	⑫ Driving switch for rotating disk
② Outlet hole of air	⑬ Switch for inside lamp of air bath
③ Motor driving apparatus for rotating disk	⑭ Switch for power supply
④ Thermometer for air bath	⑮ Switch board
⑤ Double glass window	⑯ Thermometer for sample
⑥ Door	⑰ Sample container
⑦ Handle of door	⑱ Rotating disk
⑧ Thermostat	⑲ Electric heater
⑨ Signal lamp for thermostat	⑳ Side arm
⑩ Pilot lamp for power supply	㉑ Steel plate
㉑ Switch for heater	㉒ Inlet hole of air

3. Test procedures Test procedures shall be as follows:

- (1) Clean the sample container, obtain the mass, take 20 g of the uniform sample in it, and weigh to the nearest 0.01 g.
- (2) Preliminarily keep the thermostatic air bath at  $105 \pm 1^\circ\text{C}$ , suspend a thermometer perpendicularly at the side arm attached to the shaft of the disk in a position about 20 mm inside the periphery of the disk and the lower end of the mercury bulb is situated 6 mm above the top surface of the disk.
- (3) Place the sample container containing the sample on the disk of the thermostatic air bath, then shut the door, rotate the disk at a rate of 5 to 6 revolutions per min, and leave at  $105 \pm 1^\circ\text{C}$  for 8 h. Then take out the sample container, leave it still to cool down to room temperature in a desiccator, and then weigh the mass.

4. Calculation and measurement result Calculate the evaporation loss percentage by the following formula, and express it in an average rounded off to one place of decimal from two test results.

$$W_L = \frac{W_s - W}{W_s} \times 100$$

where,  $W_L$  : evaporation loss percentage (mass %)

$W_s$  : mass of sample before test (g)

$W$  : mass of sample after test (g)

Informative reference 4. Test method for sulfated ash content

This Informative reference 4 describes the test method for sulfated ash content, and does not constitute a part of the Standard.

1. Summary of test method Burn a sample, add sulfuric acid thereto, ignite, and measure the residual content to be calculated as sulfated ash.

2. Apparatus and device The apparatus and device shall consist of the following items (1) and (2).

(1) Crucible The crucible of 15 ml capacity, made of porcelain, quartz, or platinum<sup>(1)</sup>.

Note <sup>(1)</sup> When substance such as lead, zinc, etc., which acts on platinum at high temperature is contained in the sample, the crucible made of platinum shall not be used.

(2) Muffle furnace The muffle furnace of a suitable size, capable of maintaining temperature to 600°C.

3. Reagents

(1) Sulfuric acid 10 % solution of guaranteed grade sulfuric acid specified in JIS K 8951.

(2) Methyl orange indicator 0.1 % water solution of guaranteed grade reagent specified in JIS K 8893.

(3) Ammonium carbonate Guaranteed grade reagent specified in JIS K 8613.

4. Procedure of test The procedure of the test shall be made as follows.

Heat the crucible strongly, cool it in a desiccator, and weigh after cooling. Take 2 g to 5 g of a sample into it, and obtain to the nearest 0.01 g. Heat the crucible, and burn gradually<sup>(2)</sup>. After burning, further ignite until carbon almost disappears in a muffle furnace, cool the crucible and the content, dissolve the soluble matter with a small amount of water, and cover the crucible. Insert a pipette below the cover to add a little excess of sulfuric acid with care. Warm the crucible together with its content on the hot water bath until boiling stops, and wash the attached substance on the cover into the crucible with distilled water. Drip Methyl Orange indicator in the crucible to examine the presence of free acid. Evaporate to dryness the content of the crucible, add a small amount of dry ammonium carbonate to expel the excess of sulfuric acid anhydride. Heat until the bottom of the crucible turns to faint pink color. Leave the crucible together with its content still to cool in a desiccator, and weigh.

Note <sup>(2)</sup> When the sample scatters by foaming, add 1 ml to 2 ml of ethyl alcohol before heating.

5. Calculation and result Calculate the ash content (mass %) according to the following formula, round off the average value of two measured results to one place of decimal, and take it as the test result.

$$A = \frac{W_r}{W_s} \times 100$$

where,  $A$  : ash content (mass %)

$W_r$  : mass of ash (g)

$W_s$  : mass of sample (g)

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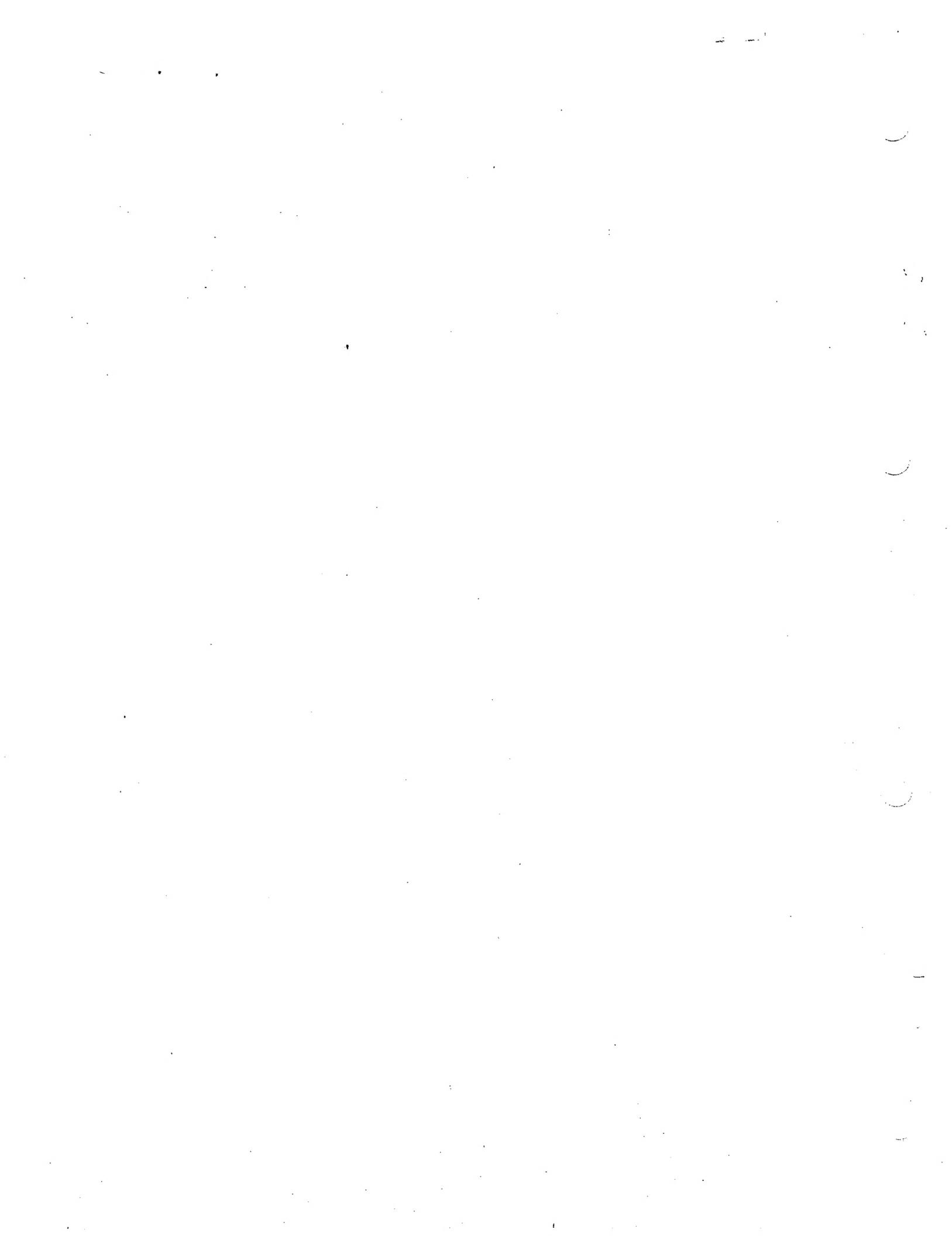
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## JIS K 6253 : 1997

### Hardness testing methods for rubber, vulcanized or thermoplastic

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ICS 83.060

**Descriptors** : vulcanized rubber, vulcanized materials, hardness testing, mechanical testing, hardness, mechanical properties of materials

**Reference number** : JIS K 6253 : 1997 (E)

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## Hardness testing methods for rubber, vulcanized or thermoplastic

**Introduction** This Japanese Industrial Standard has been prepared on the basis of the 3rd edition of ISO 48, *Rubber, vulcanized or thermoplastic—Determination of hardness (hardness between 10 IRHD and 100 IRHD)* published in 1994, and the 1st edition of ISO 7619, *Rubber—Determination of indentation hardness by means of pocket hardness meters* published in 1986, without any modification in technical contents. However, “Type E of spring type (durometer hardness)” which is not specified in the corresponding International Standards are added in this Standard.

**1 Scope** This Japanese Industrial Standard specifies the testing methods to measure hardness of vulcanized rubber and thermoplastic rubber (hereafter referred to as “vulcanized rubber”).

**Remarks 1** The standards cited in this Standard are listed as follows.

JIS K 6200 *Glossary of terms used in rubber industry*

JIS K 6250 *General rules of physical testing methods for rubber,  
vulcanized or thermoplastic*

JIS Z 8401 *Rules for rounding off of numerical values*

**2** The International Standards corresponding to this Standard are listed as follows.

ISO 48 : 1994 *Rubber, vulcanized or thermoplastic—Determination  
of hardness (hardness between 10 IRHD and 100 IRHD)*

ISO 7619 : 1986 *Rubber—Determination of indentation hardness by  
means of pocket hardness meters*

**3** The units and numerical values given in ( ) in this Standard are based on traditional units, and are appended for informative reference.

**2 Definitions** For the purposes of this Standard, the definitions given in **JIS K 6200** and **JIS K 6250**, and the following definitions apply.

(1) **international rubber hardness degree** Hardness which can be obtained through conversion into international rubber hardness degree (IRHD)<sup>(1)</sup> using the depth of indentation by a plunger when the plunger, with a ball-type lower end, is vertically impressed on the surface of a test piece with specified indenting force.

A hardness scale is chosen so that “0” represents the hardness of material having a Young’s modulus of zero and “100” represents the hardness of a material of infinite Young’s modulus, and the following conditions are fulfilled over most of normal range of hardness.

- One international rubber hardness degree always represents approximately the same proportionate difference in the Young’s modulus.
- For highly elastic rubber, the scales of international rubber hardness degree and that of type A durometer are comparable.

Note (1) IRHD International Rubber Hardness Degree

(2) **durometer hardness** The hardness given by the testing apparatus (durometer) which reads the indentation depth made by a specifically shaped indentor when it is impressed on the surface of a test piece via a spring.

(3) **IRHD pocket hardness** The hardness given by a portable pocket testing apparatus (IRHD pocket hardness meter) by which international rubber hardness degree can be conveniently obtained owing to reading the indented depth made by an indentor, with a ball-type lower end, when it impressed on the surface of a test piece via a spring.

(4) **standard hardness** The hardness obtained using the specified procedures on test pieces whose shape and dimensions satisfy the specifications, when carrying out each test.

(5) **apparent hardness** The hardness obtained either using other procedures than the specified, or on the test piece whose shape and dimensions do not satisfy the specification, when carrying out each test.

### 3 Type of test

**3.1 Outline of hardness test** There are many types of testing methods for hardness test depending on the principle of hardness measurement, range of hardness measurement, kind of testing apparatus and so on, and they are classified into standard hardness and apparent hardness by shape or dimensions of a test piece. The outline of classifying is shown in Table 1.

**Table 1** Outline of hardness tests

Principle of measurement	Range of hardness measurement	Type of testing apparatus	Testing method	Test condition for standard hardness					
				Shape	Thickness mm	Minimum distance from the edge of sample mm			
Constant-force type (international rubber hardness degree)	For high hardness (85 to 100 IRHD)	Normal size international rubber hardness meter	H method	Both upper and lower surfaces are smooth and parallel each other.	8.0 min.	9.0			
	For normal hardness (30 to 95 IRHD)	Normal size international rubber hardness meter	N method		10.0 max.	10.0			
					8.0 min.	9.0			
		Microsize international rubber hardness meter	M method		10.0 max.	10.0			
					1.5 min.	2.0			
	For low hardness (10 to 35 IRHD)	Normal size international rubber hardness meter	L method		2.5 max.				
					10.0 min.	10.0			
					15.0 max.	11.5			
					6.0 or more	12.0			
					6.0 or more	12.0			
Spring type (durometer hardness)	For high hardness (A90 or more)	Type D durometer			10.0 or more	12.0			
	For normal hardness (A10 to 90)	Type A durometer			6.0 or more	12.0			
	For low hardness (A20 or less)	Type E durometer			6.0 or more	12.0			
Spring type (IRHD pocket hardness)	For normal hardness (30 to 95 IRHD)	IRHD pocket hardness meter	P method		6.0 or more	12.0			

**3.2 Type of tests** The type of hardness tests for vulcanized rubber shall be classified as follows.

**(1) International rubber hardness test**

- (a) H method (normal size test for high hardness)
- (b) N method (normal size test for normal hardness)
- (c) M method (microsize test for normal hardness)
- (d) L method (normal size test for low hardness)

**(2) Durometer hardness test**

- (a) Type D (test for high hardness)
- (b) Type A (test for normal hardness)
- (c) Type E (test for low hardness)

**(3) IRHD pocket hardness test**

- (a) P method (for normal hardness)

**4 International rubber hardness test**

**4.1 Purpose** This test shall be carried out to measure the international rubber hardness degree of vulcanized rubber.

**4.2 Range of measurement** The measuring range of this test is decided according to the thickness and hardness of a test piece for every testing method. The measuring range of each testing method is as follows.

- (1) **H method** Formal measuring range shall be for the test piece measuring 8.0 mm to 10.0 mm in thickness and with hardness of 85 IRHD to 100 IRHD. It is permissible to test the one with 4.0 mm or more thickness and with hardness of 85 IRHD to 100 IRHD.
- (2) **N method** Formal measuring range shall be for the test piece measuring 8.0 mm to 10.0 mm in thickness and with hardness of 35 IRHD to 85 IRHD. It is permissible to test the one with 4.0 mm or more thickness and with hardness of 30 IRHD to 95 IRHD<sup>(2)</sup>.
- (3) **M method** Formal measuring range shall be for the test piece measuring 1.5 mm to 2.5 mm in thickness and with hardness of 35 IRHD to 85 IRHD. It is permissible to test the one with 1.0 mm to 4.0 mm thickness and with hardness of 30 IRHD to 95 IRHD<sup>(3)</sup>.
- (4) **L method** Formal measuring range shall be for the test piece measuring 10.0 mm to 15.0 mm in thickness and with hardness of 10 IRHD to 35 IRHD. It is permissible to test the one with 6.0 mm or more thickness and with hardness of 10 IRHD to 35 IRHD.

Notes <sup>(2)</sup> The hardness values in 85 IRHD to 95 IRHD and 30 IRHD to 35 IRHD obtained by N method do not exactly coincide with the values by H method and L method, but the discrepancy does not come into technical problem, generally speaking.

(3) The testing apparatus for M method is the one prepared by miniaturizing the testing apparatus for N method by about one-sixth to measure the test piece with thin thickness, therefore the depth of plunger indentation by M method is just one-sixth that by N method. The results given by M method are not always coincident with the results given by N method because of the surface effect of rubber or slight roughness of the surface.

#### 4.3 Testing apparatus

**4.3.1 Outline of testing apparatus** The testing apparatus is composed of a holding base for test piece by which a test piece is kept, an annular pressure foot by which the surface of a test piece is pressed, a plunger, with a ball-type lower end, set at the center of hole of pressure foot, a device for loading which gives an indenting force on a plunger to make an indentation on a test piece, a measuring device to measure depth of an indentation impressed on a test piece, and a vibrating device to lessen friction. The dimensions of main parts and the specification of force are shown in Table 2.

A thermostat may be provided for measuring a test temperature other than standard condition of laboratory.

**Table 2 Main dimensions and forces of testing apparatus**

Type of tests	Diameter of ball of plunger end mm	Face of pressure foot			Force applying at ball of plunger end		
		Diameter mm	Diameter of hole mm	Force exerted on face of pressure foot	Contact force	Indenting force	Total
H method	$1.00 \pm 0.01$	$20 \pm 1$	$6 \pm 1$				
N method	$2.50 \pm 0.01$	$20 \pm 1$	$6 \pm 1$	$8.3 \pm 1.5 \text{ N}$ ( $846 \pm 153 \text{ gf}$ )	$0.30 \pm 0.02 \text{ N}$ ( $30.6 \pm 2.0 \text{ gf}$ )	$5.40 \pm 0.01 \text{ N}$ ( $550.6 \pm 1.0 \text{ gf}$ )	$5.70 \pm 0.03 \text{ N}$ ( $581.2 \pm 3.1 \text{ gf}$ )
L method	$5.00 \pm 0.01$	$22 \pm 1$	$10 \pm 1$				
M method	$0.395 \pm 0.005$	$3.35 \pm 0.15$	$1.00 \pm 0.15$	$235 \pm 30 \text{ mN}$ ( $24.0 \pm 3.1 \text{ gf}$ ) <sup>(4)</sup>	$8.3 \pm 0.5 \text{ mN}$ ( $0.85 \pm 0.05 \text{ gf}$ )	$145 \pm 0.5 \text{ mN}$ ( $14.79 \pm 0.05 \text{ gf}$ )	$153 \pm 1 \text{ mN}$ ( $15.60 \pm 0.10 \text{ gf}$ )

Note (4) When in M method a pressure adjusting spring installed at the bottom of a test-piece holding base makes pressure adjustment, the pressure adjusting spring must be controlled to be  $(380 \pm 30) \text{ mN}$  ( $(38.7 \pm 3.1) \text{ gf}$ ) because an indenting force  $145 \text{ mN}$  ( $14.8 \text{ gf}$ ) is added during measurement.

**4.3.2 Face of pressure foot** An annular pressure foot makes rectangular to a plunger. The diameter of face of pressure foot and the diameter of the hole for a plunger are as shown in Table 2. When the force exerted on the face of pressure foot is just as shown in Table 2, the pressure impressed on the surface of test piece becomes  $(30 \pm 5) \text{ kPa}$  ( $(0.306 \pm 0.051) \text{ kgf/cm}^2$ )<sup>(5)</sup>. In order to measure the relative displacement between the face of pressure foot (upper surface of test piece) and the plunger, the face of pressure foot shall be firmly united with the measuring device of the depth of indentation.

Note (5) Some combination of all tolerances shown in Table 2 does not always give nice coincidence with the description of pressure  $(30 \pm 5) \text{ kPa}$  ( $(0.306 \pm 0.051) \text{ kgf/cm}^2$ ).

**4.3.3 Plunger** The plunger shall be vertical, and its lower end has spherical shape whose diameter shall be as shown in Table 2<sup>(6)</sup>. The lower end ball of a plunger shall be kept a little upper than the face of pressure foot before contact force is applied.

Note <sup>(6)</sup> The material of end ball shall be abrasion resistant and corrosion resistant.

When an end ball is connected with the body of plunger, the connected part must not be larger than diameter of the ball.

**4.3.4 Loading device** Loading device shall accurately apply the contact force<sup>(7)</sup> and indenting force<sup>(8)</sup> specified in Table 2 to the end ball of a plunger.

Notes <sup>(7)</sup> Contact force means the force causing the end ball of a plunger to contact with surface of a test piece.

<sup>(8)</sup> Indenting force means the force to impress the end ball of a plunger into test piece after making contact.

**4.3.5 Measuring device of indented depth** The measuring device for indented depth shall be capable of measuring indented depth of a plunger when indenting force is applied to a plunger, by which the indented depth or IRHD shall be directly read<sup>(9)</sup>. The conversion from indented depth to IRHD can be done through Table 3, Table 4 and Table 5<sup>(10)</sup>.

Notes <sup>(9)</sup> For the measuring device of indented depth, any of mechanical, optical, or electrical, is serviceable.

<sup>(10)</sup> Table 3 is for the conversion of H method, and Table 4 for N method. In case of M method, convert after making the indented depth shown in Table 4 one-sixth. Table 5 is the conversion table for L method.

**4.3.6 Vibrating device** To overcome minute friction, it is preferable to install a vibrating device like an electric buzzer by which a testing apparatus is suitably vibrated. It can be eliminated if friction is completely removed.

**4.3.7 Thermostat** The thermostat is needed when the test temperature other than standard condition of laboratory is employed for measuring hardness. The thermostat must keep the specified temperature in the tolerance of  $\pm 2$  °C. The annular foot with pressure face at lower end and a plunger shall penetrate through the upper part of the thermostat.

The part through which the plunger penetrates shall be made of the material with small thermal conductivity. The sensor for temperature measurement shall be installed at holding place of test piece or its vicinity, in the thermostat.

**Table 3** Conversion table from indented depth ( $D$ ) of a plunger to international rubber hardness degree (IRHD) (H method)

$D$ mm	International rubber hardness degree IRHD	$D$ mm	International rubber hardness degree IRHD	$D$ mm	International rubber hardness degree IRHD
0.00	100.0	0.15	97.3	0.30	91.1
0.01	100.0	0.16	97.0	0.31	90.7
0.02	100.0	0.17	96.6	0.32	90.2
0.03	99.9	0.18	96.2	0.33	89.7
0.04	99.9	0.19	95.8	0.34	89.3
0.05	99.8	0.20	95.4	0.35	88.8
0.06	99.6	0.21	95.0	0.36	88.4
0.07	99.5	0.22	94.6	0.37	87.9
0.08	99.3	0.23	94.2	0.38	87.5
0.09	99.1	0.24	93.8	0.39	87.0
0.10	98.8	0.25	93.4	0.40	86.6
0.11	98.6	0.26	92.9	0.41	86.1
0.12	98.3	0.27	92.5	0.42	85.7
0.13	98.0	0.28	92.0	0.43	85.3
0.14	97.6	0.29	91.6	0.44	84.8

**Table 4** Conversion table from indented depth ( $D$ ) of a plunger to international rubber hardness degree (IRHD) (N method)

$D$ mm	International rubber hardness degree IRHD						
0.00	100.0	0.45	73.9	0.90	52.3	1.35	38.9
0.01	100.0	0.46	73.3	0.91	52.0	1.36	38.7
0.02	99.9	0.47	72.7	0.92	51.6	1.37	38.4
0.03	99.8	0.48	72.2	0.93	51.2	1.38	38.2
0.04	99.6	0.49	71.6	0.94	50.9	1.39	38.0
0.05	99.3	0.50	71.0	0.95	50.5	1.40	37.8
0.06	99.0	0.51	70.4	0.96	50.2	1.41	37.5
0.07	98.6	0.52	69.8	0.97	49.8	1.42	37.3
0.08	98.1	0.53	69.3	0.98	49.5	1.43	37.1
0.09	97.7	0.54	68.7	0.99	49.1	1.44	36.9
0.10	97.1	0.55	68.2	1.00	48.8	1.45	36.7
0.11	96.5	0.56	67.6	1.01	48.5	1.46	36.5
0.12	95.9	0.57	67.1	1.02	48.1	1.47	36.2
0.13	95.3	0.58	66.6	1.03	47.8	1.48	36.0
0.14	94.7	0.59	66.0	1.04	47.5	1.49	35.8
0.15	94.0	0.60	65.5	1.05	47.1	1.50	35.6
0.16	93.4	0.61	65.0	1.06	46.8	1.51	35.4
0.17	92.7	0.62	64.5	1.07	46.5	1.52	35.2
0.18	92.0	0.63	64.0	1.08	46.2	1.53	35.0
0.19	91.3	0.64	63.5	1.09	45.9	1.54	34.8
0.20	90.6	0.65	63.0	1.10	45.6	1.55	34.6
0.21	89.8	0.66	62.5	1.11	45.3	1.56	34.4
0.22	89.2	0.67	62.0	1.12	45.0	1.57	34.2
0.23	88.5	0.68	61.5	1.13	44.7	1.58	34.0
0.24	87.8	0.69	61.1	1.14	44.4	1.59	33.8
0.25	87.1	0.70	60.6	1.15	44.1	1.60	33.6
0.26	86.4	0.71	60.1	1.16	43.8	1.61	33.4
0.27	85.7	0.72	59.7	1.17	43.5	1.62	33.2
0.28	85.0	0.73	59.2	1.18	43.3	1.63	33.0
0.29	84.3	0.74	58.8	1.19	43.0	1.64	32.8
0.30	83.6	0.75	58.3	1.20	42.7	1.65	32.6
0.31	82.9	0.76	57.9	1.21	42.5	1.66	32.4
0.32	82.2	0.77	57.5	1.22	42.2	1.67	32.3
0.33	81.5	0.78	57.0	1.23	41.9	1.68	32.1
0.34	80.9	0.79	56.6	1.24	41.7	1.69	31.9
0.35	80.2	0.80	56.2	1.25	41.4	1.70	31.7
0.36	79.5	0.81	55.8	1.26	41.1	1.71	31.6
0.37	78.9	0.82	55.4	1.27	40.9	1.72	31.4
0.38	78.2	0.83	55.0	1.28	40.6	1.73	31.2
0.39	77.6	0.84	54.6	1.29	40.4	1.74	31.1
0.40	77.0	0.85	54.2	1.30	40.1	1.75	30.9
0.41	76.4	0.86	53.8	1.31	39.9	1.76	30.7
0.42	75.8	0.87	53.4	1.32	39.6	1.77	30.5
0.43	75.2	0.88	53.0	1.33	39.4	1.78	30.4
0.44	74.5	0.89	52.7	1.34	39.1	1.79	30.2
						1.80	30.0

**Table 5** Conversion table from indented depth ( $D$ ) of a plunger to international rubber hardness degree (IRHD) (L method)

$D$ mm	International rubber hardness degree IRHD	$D$ mm	International rubber hardness degree IRHD	$D$ mm	International rubber hardness degree IRHD
1.10	34.9	1.80	21.3	2.50	14.1
1.12	34.4	1.82	21.1	2.52	14.0
1.14	33.9	1.84	20.8	2.54	13.8
1.16	33.4	1.86	20.6	2.56	13.7
1.18	32.9	1.88	20.3	2.58	13.5
1.20	32.4	1.90	20.1	2.60	13.4
1.22	31.9	1.92	19.8	2.62	13.3
1.24	31.4	1.94	19.6	2.64	13.1
1.26	30.9	1.96	19.4	2.66	13.0
1.28	30.4	1.98	19.2	2.68	12.8
1.30	30.0	2.00	18.9	2.70	12.7
1.32	29.6	2.02	18.7	2.72	12.6
1.34	29.2	2.04	18.5	2.74	12.5
1.36	28.8	2.06	18.3	2.76	12.3
1.38	28.4	2.08	18.0	2.78	12.2
1.40	28.0	2.10	17.8	2.80	12.1
1.42	27.6	2.12	17.6	2.82	12.0
1.44	27.2	2.14	17.4	2.84	11.8
1.46	26.8	2.16	17.2	2.86	11.7
1.48	26.4	2.18	17.0	2.88	11.6
1.50	26.1	2.20	16.8	2.90	11.5
1.52	25.7	2.22	16.6	2.92	11.4
1.54	25.4	2.24	16.4	2.94	11.3
1.56	25.0	2.26	16.2	2.96	11.2
1.58	24.7	2.28	16.0	2.98	11.1
1.60	24.4	2.30	15.8	3.00	11.0
1.62	24.1	2.32	15.6	3.02	10.9
1.64	23.8	2.34	15.4	3.04	10.8
1.66	23.5	2.36	15.3	3.06	10.6
1.68	23.1	2.38	15.1	3.08	10.5
1.70	22.8	2.40	14.9	3.10	10.4
1.72	22.5	2.42	14.8	3.12	10.3
1.74	22.2	2.44	14.6	3.14	10.2
1.76	21.9	2.46	14.4	3.16	10.1
1.78	21.6	2.48	14.3	3.18	9.9

#### 4.4 Test piece

**4.4.1 Shape of test pieces** Both surfaces of a test piece shall be smoothly flat and parallel each other<sup>(11)</sup>. This test has been supposed to compare the test pieces having the same thickness.

**Note (11)** The surface such as unsmoothed, curved, or rough, does not give satisfactory results. For specially formed surface, however, such as rubber roll, this method can be applied.

The international rubber hardness testing method for curved test piece is shown in Informative reference.

#### 4.4.2 Thickness

(1) **H method and N method** The standard thickness of a test piece is 8.0 mm to 10.0 mm, but to get necessary thickness, it is permissible to pile smooth and parallel test pieces. Provided that the thickness of test pieces before piling shall be 2 mm or more, and 3 or more test pieces cannot be piled up. Even when nonstandard test piece other than above<sup>(12)</sup> is to be adopted, the thickness of the test piece must be 4.0 mm or more.

(2) **L method** The standard thickness of a test piece is 10.0 mm to 15.0 mm, but to get necessary thickness, it is permissible to pile smooth and parallel test pieces. Provided that the thickness of test pieces before piling shall be 2 mm or more, and 3 or more test pieces cannot be piled up. Even when nonstandard test piece other than above<sup>(12)</sup> is to be adopted, the thickness of the test piece must be 6.0 mm or more.

(3) **M method** The standard thickness of a test piece is  $(2.0 \pm 0.5)$  mm. Even when nonstandard test piece other than above<sup>(12)</sup> is to be adopted, the thickness of the test piece must be 1.0 mm or more.

**Note (12)** The measured value resulted from nonstandard test piece, is not generally coincident with the measured value by standard test piece.

#### 4.4.3 Lateral dimensions

(1) **H method, N method, and L method** The lateral dimension of a test piece shall be large enough to measure at the point which is apart from edge of the test piece by at least the distance shown in Table 6.

**Table 6** Minimum distance of point for hardness measurement (point of end ball of plunger) from test-piece edge

Unit: mm

Thickness of a test piece	Minimum distance of point for hardness measurement from test-piece edge
4.0	7.0
6.0	8.0
8.0	9.0
10.0	10.0
15.0	11.5
25.0	13.0

(2) **M method** The lateral dimension of a test piece shall be large enough to measure at the point which is apart from edge of the test piece by at least 2.0 mm. When the test piece, with the thickness of 4.0 mm or more, which is not eligible for N method because of small lateral dimension or of not having large smooth area, is to be tested by M method, carry out test at the point apart from edge of the test piece as far as possible.

**4.4.4 Sampling and preparation of test pieces** The sampling and preparation of test pieces shall principally follow 6.5 of **JIS K 6250**.

**4.4.5 Selection of test pieces** The test pieces which contain alien matters, bubbles, or flaws shall not be used for tests.

#### **4.5 Testing method**

**4.5.1 Testing conditions** Testing conditions shall be as follows.

- (1) The standard conditions of a laboratory shall follow **6.1 of JIS K 6250**.
- (2) Storing of sample and test pieces shall follow **6.2 of JIS K 6250**.
- (3) The standard conditions of test pieces shall follow **6.3 of JIS K 6250**.

**4.5.2 Procedures** Sprinkle slightly talc on upper and back surfaces of a test piece to lessen friction between the end ball of a plunger and surface of a test piece. Place the test piece on the holding base of a test piece. Make the face of pressure foot touch with the surface of the test piece.

- (1) When the scale is graduated with IRHD, apply contact force to the plunger for 5 s, and adjust the scale to be 100. Then, apply indenting force for 30 s, and read directly hardness by IRHD.
- (2) When the scale is graduated with indented depth, apply contact force to the plunger for 5 s, and read the scale. Then, apply indenting force for 30 s, and read the scale. Calculate the difference between indentation by contact force and that by indenting force, and make this the indented depth  $D$ . Convert the value of  $D$  into IRHD making use of Table 3, Table 4, and Table 5.

While applying force, the slight vibration may be applied on the testing apparatus by a vibrating device to overcome the friction. Carry out measurements at 3 or 5 new points on a test piece at every measurement.

**4.6 Arrangement of test results** Round off the median of 3 or 5 measurements to whole number according to **JIS Z 8401**, and mark the sign IRHD after it. In case of standard hardness, after it mark "/" together with letter "S", and then mark "/" with sign as H, N, M, or L, which means testing method. In case of apparent hardness, after sign of IRHD mark "/" together with sign as H, N, M, or L, which means testing method.

**Example 1** 50 IRHD/S/N: means that standard test piece is measured by N method of international rubber hardness test, and standard hardness is 50 IRHD.

**Example 2 50 IRHD/M:** means that nonstandard test piece is measured by M method of international rubber hardness test, and apparent hardness is 50 IRHD.

**4.7 Record** On test result, the following items shall be recorded.

- (1) Test result
- (2) Shape and dimensions of test piece (whether standard test piece or nonstandard one; in case of nonstandard, whether curved surface or not; and in case of piled one, the number of piled pieces and its thickness)
- (3) Sampling and preparation methods of test pieces
- (4) Test temperature
- (5) Other items specially needed

**5 Durometer hardness test**

**5.1 Purpose** This test shall be carried out to measure durometer hardness of vulcanized rubber.

**5.2 Range of measurement** The measuring range of this test is decided according to the hardness of test piece at every testing method. The measuring range of each testing method is as follows.

- (1) **Type D durometer** The measuring range of type D durometer hardness is the range over A90 by type A durometer. When less than D20, measure by type A durometer.
- (2) **Type A durometer** The measuring range of type A durometer hardness is from A10 to A90, and when over A90, measure by type D durometer. When less than A20, measure by type E durometer.
- (3) **Type E durometer** The measuring range of type E durometer hardness is the range of less than A20 by type A durometer.

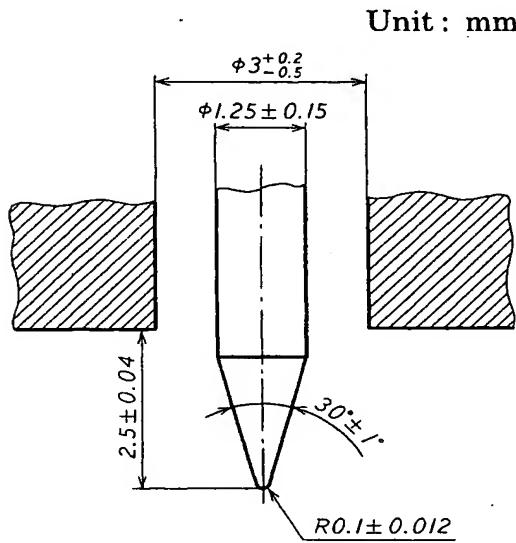
**5.3 Testing apparatus**

**5.3.1 Outline of testing apparatus** The testing apparatus is composed of the face of pressure foot by which the surface of a test piece is pressed, indentor which protrudes from a central hole of face of pressure foot by action of a spring, and the graduation which indicates the distance (indenting depth) of indentor rejected by rubber cushion and which represents hardness itself.

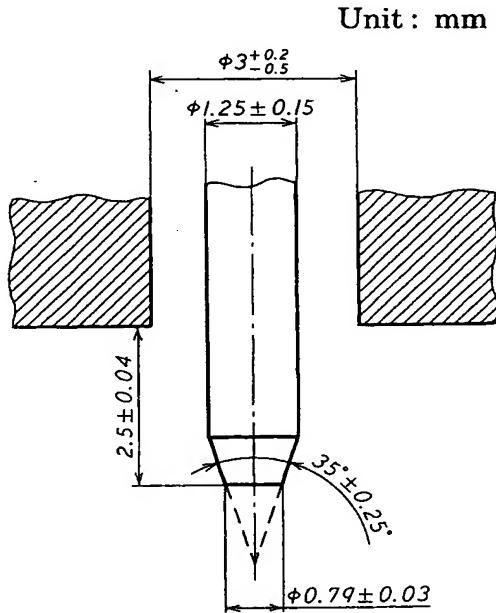
**5.3.2 Face of pressure foot** The face of pressure foot is perpendicular to the indentor, and its center has a hole for the indentor. The diameter of the hole, in case of type D and type A durometer, is  $3.0^{+0.2}_{-0.5}$  mm, and in case of type E durometer,  $(5.4 \pm 0.2)$  mm.

On the face of pressure foot, the distance from any place of its outer edge to the center of an indentor shall be, in case of type D and type A durometer, 6 mm or more, and in case of type E durometer, 7 mm or more.

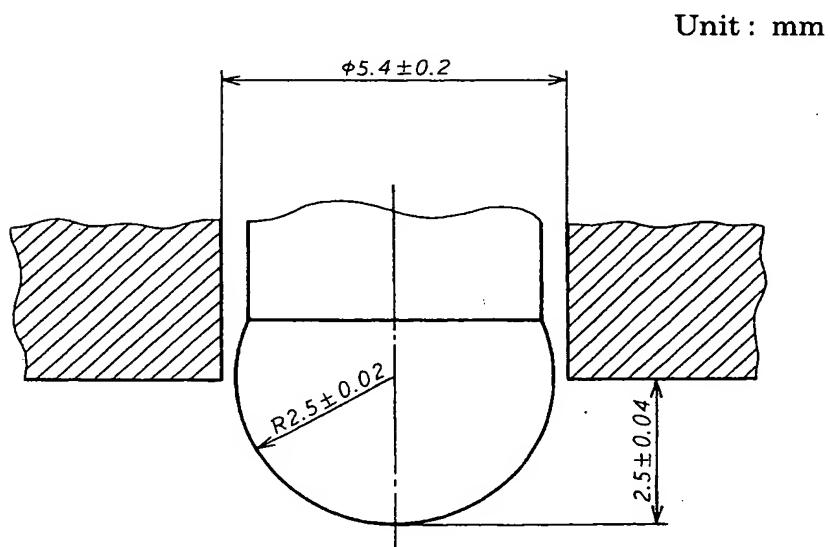
**5.3.3 Indentor** The material of indentor shall be abrasion resistant and corrosion resistant, and it shall be accurately fixed at center of the hole of face of pressure foot. Its shape and dimensions are indicated in Fig. 1 for type D durometer, in Fig. 2 for type A durometer, and in Fig. 3 for type E durometer.



**Fig. 1** Indentor for type D durometer



**Fig. 2** Indentor for type A durometer



**Fig. 3** Indentor for type E durometer

**5.3.4 Scale** When the scale indicates 0 (full protrusion), the point of the indentor shall protrude by  $(2.50 \pm 0.04)$  mm beyond the face of the pressure foot.

When the scale indicates 100 (nil protrusion), the face of the pressure foot is in firm contact with a flat piece of glass, i.e. the point of the indentor shall be positioned on the same plane with the face of the pressure foot. The scale shall be graduated with equal intervals in the range between 0 to 100.

**5.3.5 Spring** There must be the following relation between the force of spring and the scale, that is, the durometer hardness.

(1) **Type D durometer**

$$W_D = 444.5 H_D \{w_D = 45.33 H_D\}$$

where,  $W_D$  : force of spring of type D durometer (mN)

$w_D$  : force of spring of type D durometer (gf)

$H_D$  : hardness of type D durometer

(2) **Type A and type E durometer**

$$W_A = 550 + 75 H_A \{w_A = 56.1 + 7.65 H_A\}$$

where,  $W_A$  : force of spring of type A or type E durometer (mN)

$w_A$  : force of spring of type A or type E durometer (gf)

$H_A$  : hardness of type A or type E durometer

The tolerance of force shall be, in case of type D durometer,  $\pm 440$  mN  $\{\pm 44.9$  gf $\}$ , and in case of type A and type E durometer,  $\pm 80$  mN  $\{\pm 8.16$  gf $\}$ .

**5.3.6 Calibration of spring** Hold vertically the end point of indentor of a durometer on a balance not to give any interference between the balance and face of pressure foot, via a spacer (see Fig. 4). The cylindrical spacer with 2.5 mm height, in case of type D and type A durometer, measuring 1.25 mm in diameter, and in case of type E durometer, measuring 3 mm in diameter, has a wineglass shape where an indentor is to touch, in order to smoothly receive the end point of the indentor. Place a tare on the balance against the weight of the spacer. Place counterweight to get suitable scale, and confirm that the force (mN) shown here stays within the tolerance of specified force in 5.3.5. Carry out the above calibration using suitable scale interval.

The calibration of spring of a durometer may be done with an electrobalance other than chemical balance shown in Fig. 4. In this case, the measuring sensitivity of the force at end point of an indentor shall be, in case of type D durometer, 44 mN (4.5 gf) or less, and in case of type A and type E durometer, 8 mN (0.82 gf) or less.

The following method is permissible; place upside down the durometer, and directly apply the load on its indentor by counterweight. Provided that the correction about the mass of parts inside of the durometer shall be considered to prevent the discrepancy between this method and the method by Fig. 4. In this case, the accuracy on the mass of counterweight shall be  $\pm 4.5$  g or less in case of type D durometer and  $\pm 0.82$  g or less in case of type A and type E durometer.

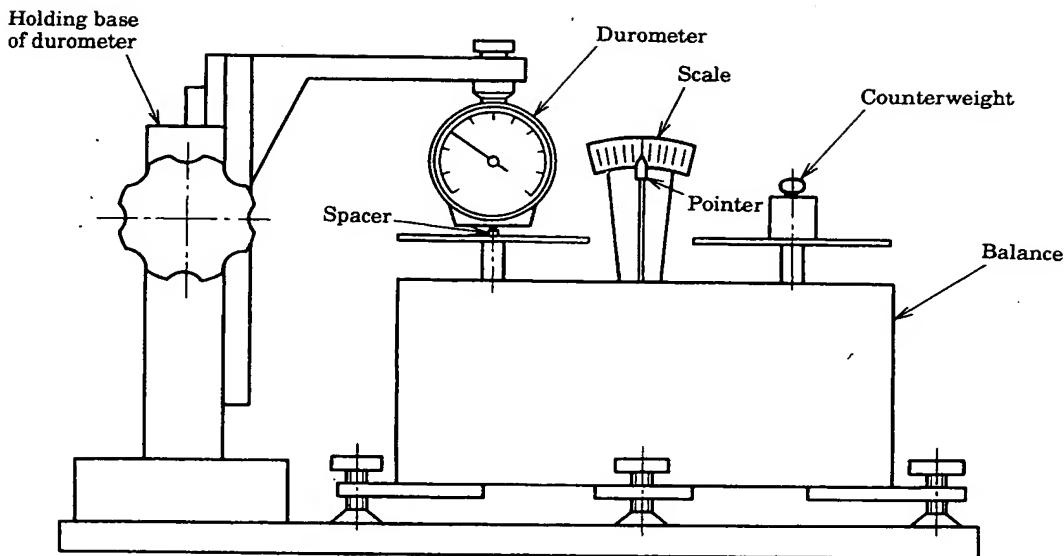


Fig. 4 Example of calibration apparatus of spring

#### 5.4 Test piece

**5.4.1 Shape and dimensions of test pieces** The thickness of a test piece for type D and type A durometer is 6 mm or more. When it is less than 6 mm, pile them to make 6 mm or more for measurement. The thickness of a test piece for type E durometer is 10 mm or more, and in case of less than 10 mm, pile them to make 10 mm or more. The number of test pieces to pile shall be at most 3, and each of them shall have 2 mm or more thickness. The test result brought by piled up test piece doesn't generally coincide with the result by solid test piece<sup>(13)</sup>. The lateral size of test piece shall be large enough to measure at the point where the end point of an indentor is apart 12 mm or more from the edge of the test piece.

Furthermore, the test piece shall have smooth surface spacious enough to make close contact with face of pressure foot of a durometer<sup>(14)</sup>.

Notes (13) To make comparison, it is necessary to use the test piece which has the same number for piling and the same thickness.

(14) The surface such as unsmoothed, curved, or rough, does not give satisfactory results. For specially formed surface, however, such as rubber roll, this method can be applied. In this case, the applicable limit of the durometer shall be definitely confirmed.

**5.4.2 Sampling and preparation of test pieces** The sampling and preparation of test pieces shall follow 6.5 of JIS K 6250.

**5.4.3 Selection of test pieces** The test pieces which contain alien matters, bubbles, or flaws shall not be used for test.

#### 5.5 Testing method

##### 5.5.1 Testing conditions

Testing conditions shall be as follows.

- (1) The standard conditions of a laboratory shall follow 6.1 of JIS K 6250.

- (2) Storing of sample and test pieces shall follow 6.2 of **JIS K 6250**.
- (3) The standard conditions of test pieces shall follow 6.3 of **JIS K 6250**.

**5.5.2 Procedures** Place a test piece on a rigid, hard, and flat surface. Set a durometer so as to make an indentor rectangular to the target surface of a test piece. Contact closely as swiftly as possible the face of pressure foot with the target surface of the test piece without giving a impact, and read the scale within 1 s, to find the hardness of the test piece<sup>(15)</sup>. But the agreement between the parties concerned with delivery may permit to read when a definite time passed after close contacting between them. The end point of the indentor of a durometer must be apart 12 mm or more from the edge of the test piece. Unless otherwise specified, the duration from close contacting to the finish of reading shall be recorded. The measuring points shall be 5, which are apart at least 6 mm each other, and carry out measurements 5 times on these points. When hardness shown by type A durometer is over A90, employ a type D durometer. When the hardness shown by type D durometer is less than D20, employ a type A durometer. If the hardness by type A durometer is less than A10, result is inaccurate, so don't record it.

When the hardness by a type A durometer is less A20, measure it with a type E durometer.

**Note (15)** In order to get a good repeatability, the holding base for durometer may be used by which the durometer is vertically kept and target surface and indentor get right angle each other before measurement. In this case, it is recommended that the mass imposed on the pressing surface is 5.0 kg for type D durometer, and 1.0 kg for both type A and type E durometer.

**5.6 Arrangement of test results** Round off the median of 5 measurements to whole number according to **JIS Z 8401**, and mark sign D in case of type D durometer, sign A in case of type A durometer, and sign E in case of type E durometer, just before the rounded value. When the value was read when definite time passed after close contacting, mark sign "/" and then record the duration (s). When it is standard hardness, the above is followed by "/" and then by sign S.

**Example 1** D85/15/S: means that standard test piece is measured by type D durometer hardness test, and the reading on standard hardness is 85 when 15 s passed after close contacting of face of pressure foot.

**Example 2** A45/S: means that standard test piece is measured by type A durometer hardness test, and the reading on standard hardness is 45 within 1 s after close contacting of face of pressure foot.

**Example 3** A45/15: means that nonstandard test piece is measured by type A durometer hardness test, and the reading on apparent hardness is 45 when 15 s passed after close contacting of face of pressure foot.

**Example 4** E60: means that nonstandard test piece is measured by type E durometer hardness test, and the reading on apparent hardness is 60 within 1 s after close contacting of face of pressure foot.

## **5.7 Record** On test result, the following items shall be recorded.

- (1) Test result

- (2) Shape and dimensions of test piece (whether standard test piece or nonstandard test piece; in case of piled up test piece, the number of piled pieces, and its thickness)
- (3) Sampling and preparation methods of test pieces
- (4) Other items specially needed

## 6 IRHD pocket hardness test

**6.1 Purpose** This test shall be carried out to measure the international rubber hardness degree of vulcanized rubber by IRHD pocket hardness meter, and abbreviated P method.

### 6.2 Testing apparatus

**6.2.1 Outline of testing apparatus** The testing apparatus is composed of a face of pressure foot to press the surface of a test piece, indentor which protrudes from a central hole of face of pressure foot by action of a spring, and a mechanism indicating the protruded length of the indentor.

**6.2.2 Face of pressure foot** The face of pressure foot, measuring  $(20 \pm 2.5)$  mm sided square, has a hole with 2.0 mm to 3.0 mm diameter at its center.

**6.2.3 Indentor** The end of the indentor shall make a hemisphere with 1.55 mm to 1.60 mm diameter.

**6.2.4 Indicating mechanism** The indicating mechanism shows the protruded length of an indentor from face of pressure foot, and it shall have been calibrated to read directly the international rubber hardness degree by IRHD. When the longest protruded length of 1.65 mm is given, it must show 28 IRHD, and when the face of pressure foot is let contact with a flat glass, that is, no protruded, it must show 100 IRHD.

**6.2.5 Spring** Spring can apply constant force of  $(2.65 \pm 0.15)$  N  $((270.3 \pm 15.3)$  gf) to an indentor in the range from 28 IRHD to 100 IRHD.

**6.2.6 Calibration of hardness meter** IRHD pocket hardness meter shall be calibrated and adjusted using a standard rubber block whose international rubber hardness degree has been known. Only when the standard rubber block cannot be used, it is preferably calibrated with mechanical method.

Press the IRHD pocket hardness meter on a flat glass plate, and adjust the scale to get 100 IRHD. Making use of a set of standard rubber blocks from 30 IRHD to 90 IRHD, calibrate IRHD pocket hardness meter. The set of standard rubber blocks is stored in a container with a suitable cover after being sprinkled with talc powder, in order to prevent the influences by light, heat, oil, or grease. It consists of at least 6 test pieces. These standard blocks must be calibrated with the international rubber hardness test specified in 4 at intervals not exceeding six months. It is advisable that the IRHD pocket hardness meter, which is used daily, is calibrated at least once a week with standard rubber block.

**Remarks :** When IRHD pocket hardness meter is calibrated with mechanical method or adjusted, the instruction manual issued by the manufacturer shall be depended.

### 6.3 Test piece

**6.3.1 Shape and dimensions of test pieces** The thickness of a test piece shall be 6 mm or more. When it is less than 6 mm, the test piece which was prepared by piling up to 6 mm or more can be used, but the number of piling up shall be 3 or less, and each of them shall have 2 mm or more thickness. The test result comes from piled test piece does not usually coincide with the test result by solid test piece<sup>(18)</sup>. The lateral dimension of a test piece shall be large enough to measure at the point where the end point of an indentor is apart 12 mm or more from the edge of the test piece.

Test pieces shall have flat surface which is spacious to closely contact with the face of pressure foot of a hardness meter<sup>(16)</sup>.

Note (16) The surface such as unsmoothed, curved, or rough, does not give satisfactory results. For specially formed surface, however, such as rubber roll, this method can be applied. In this case, the applicable limit of the IRHD pocket hardness meter shall be definitely confirmed.

**6.3.2 Sampling and preparation of test pieces** The sampling and preparation of test pieces shall follow 6.5 of JIS K 6250.

**6.3.3 Selection of test pieces** The test pieces which contain alien matters, bubbles, or flaws shall not be used for test.

### 6.4 Testing method

**6.4.1 Testing conditions** Testing conditions shall be as follows.

- (1) The standard conditions of a laboratory shall follow 6.1 of JIS K 6250.
- (2) Storing of sample and test pieces shall follow 6.2 of JIS K 6250.
- (3) The standard conditions of test pieces shall follow 6.3 of JIS K 6250.

**6.4.2 Procedures** Place a test piece on a rigid, hard, and flat surface. Set an IRHD pocket hardness meter so as to make an indentor rectangular to the target surface of a test piece. Contact closely as swiftly as possible the face of pressure foot with the target surface of the test piece without giving a impact, and read the scale within 1 s, to find the hardness of the test piece. The end point of the indentor of an IRHD pocket hardness meter must be apart 12 mm or more from the edge of the test piece. Unless otherwise specified, read the value within 1 s after close contacting, but if the reading after special duration is specified, follow that specification. In this case, the duration from close contacting to the finish of reading shall be recorded. The measuring points shall be 5, which are apart at least 6 mm each other, and carry out measurements 5 times on these points.

**6.5 Arrangement of test results** Round off the median of 5 measurements to whole number according to JIS Z 8401, then mark sign IRHD after the value, and in case of standard hardness, after the value mark sign "/", then sign S, then again sign "/" and last sign P which means testing method. In case of apparent hardness, mark sign "/" after sign IRHD, then mark sign P which means testing method.

**Example 1** 50 IRHD/S/P: means that standard test piece is measured by IRHD pocket hardness meter, and the standard hardness is 50 IRHD.

**Example 2** 50 IRHD/P: means that nonstandard test piece is measured by IRHD pocket hardness meter, and the apparent hardness is 50 IRHD.

**6.6 Record** On test result, the following items shall be recorded.

- (1) Test result
- (2) Shape and dimensions of test piece (whether standard test piece or nonstandard test piece; in case of piled up test piece, the number of piled pieces, and its thickness)
- (3) Sampling and preparation methods of test pieces
- (4) Other items specially needed

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**Related standards :**

ISO 7267/1 : 1986 *Rubber-covered rollers—Determination of apparent hardness—Part 1 : IRHD method*

ISO 7267/2 : 1986 *Rubber-covered rollers—Determination of apparent hardness—Part 2 : Shore-type durometer method*

**Informative reference**  
International rubber hardness testing method for curved test piece

**Introduction** This Informative reference states the international rubber hardness testing method for curved test piece, and does not make a part of Standard.

**1 Purpose** This test shall be carried out to measure international rubber hardness degree of a test piece of vulcanized rubber whose target surface makes a curved surface. The measured values obtained by this method are always treated as an apparent hardness.

**Remarks :** The standards cited in this Informative reference are listed as follows.

ISO 48 : 1994 *Rubber, vulcanized or thermoplastic—Determination of hardness (hardness between 10 IRHD and 100 IRHD)*

ISO 7267/1 : 1986 *Rubber-covered rollers—Determination of apparent hardness—Part 1 : IRHD method*

ISO 7267/2 : 1986 *Rubber-covered rollers—Determination of apparent hardness—Part 2 : Shore-type durometer method*

**2 Type of testing method**

- (1) CH method (normal size curved surface test for high hardness)
- (2) CN method (normal size curved surface test for normal hardness)
- (3) CM method (microsize curved surface test for normal hardness)
- (4) CL method (normal size curved surface test for low hardness)

**3 Scope** CH method, CN method, CM method, and CL method are the modified H method, N method, M method, and L method for the purpose of making them applicable to the test piece whose target surface is curved, and there are the following two cases<sup>(1)</sup>.

- (1) Test piece or sample is large enough to place the hardness testing apparatus on it.
- (2) Test piece or sample is so small that it must be placed on a holding base together with a hardness testing apparatus. The case where the sample is put on a flat sample base which makes one body with a testing apparatus, is included in this case.

**Note** (1) Generally, these tests are carried out directly on products, so that the thickness of rubber is not constant, and in many cases, the lateral distance from the end ball of a plunger to the edge of sample is smaller than the smallest distance shown in 4.4.3 in the body of this Standard, and the influence owing to the distance from the edge is not negligible.

Therefore, the measured values resulted from these methods don't coincide with the values obtained by the measurements of the plate-type test pieces with flat parallel surfaces and the same thickness as that of standard test pieces or products which are specified in H method, N method, M method and L method.

This means that, the results obtained by measuring curved surface are the peculiar measurements which are applicable only to the test pieces or the products having special shape and special dimensions and further being kept in special method. In extreme case, these measured values show discrepancy of 10 IRHD from the standard hardness. The measured values on the surface buffed to eliminate covered cloth or treated specially, shows a little difference value from the value on flat surface which has been finished with molding.

#### 4 Testing apparatus

**4.1 General matters** Basically, testing apparatus follows 4.3 of the body of this Standard, but the following gives difference.

**4.2 Testing apparatus for cylindrical surface of 50 mm or more radius** As shown in Informative reference Fig. 1, the bottom base of the testing apparatus has a hole through which annular pressure foot can penetrate, for the measurement even when sample is put under the base.

There are two cylindrical surfaces which are parallel each other under the base, and these are parallel to the horizontal surface of the base. The diameter of these cylinders and the distance between them shall be suitable for setting up testing apparatus on the target curved surface of sample. Alternatively, the base, on which adjustable legs with universal joints are attached to comply with the target curved surface, may be used.

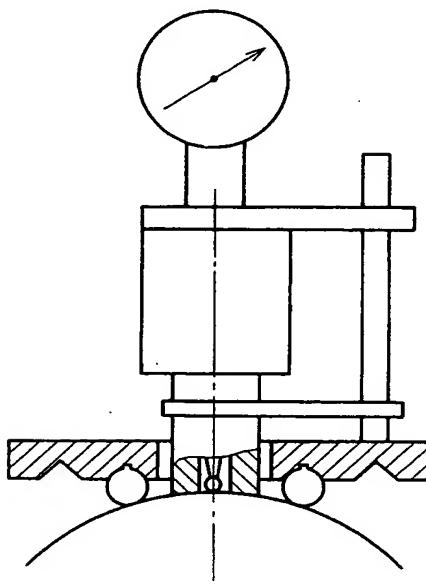
**4.3 Testing apparatus for two-way curved surface of 50 mm or more radius** The testing apparatus with adjustable legs with universal joints shown in 4.2 can be used.

**4.4 Testing apparatus for cylindrical surface and two-way curved surface of 4 mm to 50 mm radius** When target surface is too small to set a testing apparatus on it, as shown in Informative reference Fig. 2, fix test piece or sample using a special jig, V-block, or the like, and set the plunger to be perpendicular onto the target surface. When a small test piece is fixed on a sample table, wax may be used<sup>(2)(3)</sup>.

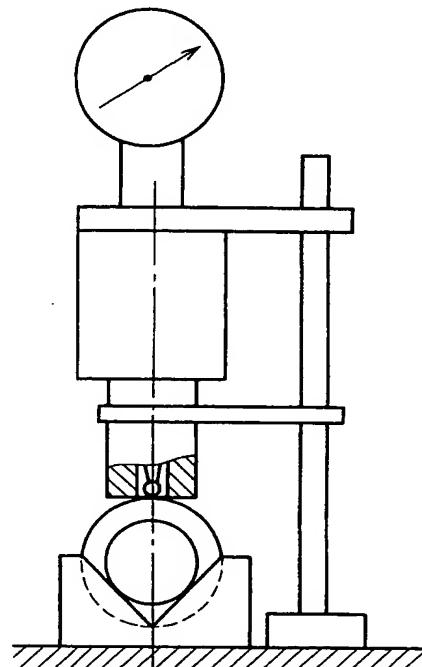
Notes (2) The testing apparatus for M method shall be generally used only for the test piece whose thickness is 4 mm or less.

(3) The testing apparatus for M method, whose sample table is forced up owing to the action of a spring, is not suitable for the large-sized test piece or sample having curved surface with large radius.

**4.5 Testing apparatus for small type O-ring and curved sample of 4 mm or less radius** In these cases, hold a test piece on the table of testing apparatus using a suitable jig, block, wax, or the like. Carry out measurement using a testing apparatus of M method. The test piece having the minimum radius of 0.8 mm or less cannot be measured.



**Informative reference Fig. 1**  
Example of setting a testing apparatus for sample with large diameter



**Informative reference Fig. 2**  
Example of setting a testing apparatus for sample with small diameter

## 5 Test pieces

**5.1 General matters** The test pieces for CH method, CN method, CM method, and CL method are the products or the pieces prepared by cutting the products. The bottom side of the test piece which has been cut out shall be held with suitable method. In case of the target surface is covered with cloth, it must be buffed before testing. In order to recover it from the influence by buffing, allow it to stand for 16 h or more under standard condition of laboratory, and then carry out conditioning under standard condition according to (3) of 4.5.1 in the body of this Standard. This duration may be included in the duration for recovering.

**5.2 Sampling and preparation of test pieces** The sampling and preparation of test pieces shall follow 4.4.4 in the body of this Standard.

**5.3 Selection of test pieces** The selection of test pieces shall follow 4.4.5 in the body of this Standard.

**6 Testing method** The testing method shall follow 4.5 in the body of this Standard.

**7 Arrangement of test results** Round off the median of 3 or 5 measurements to whole number according to JIS Z 8401, and then mark sign IRHD after the value. After that, mark sign "/", and then mark CH, CN, CM, or CL which means testing method.

Example: 50 IRHD/CM: means that a curved test piece is measured by CM method of international rubber hardness curved-surface test, and the hardness is 50 IRHD.

**8 Record** On test result, the following items shall be recorded.

- (1) Test result
- (2) Shape and dimensions of test pieces
- (3) Sampling and preparation methods of test piece
- (4) Test temperature
- (5) Other items specially needed

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## Designation: 310/75

of National Lubricating Grease Institute (NLGI) consistency numbers 0 to 4 and is intended for use only where the size of the sample prevents the use of Test Method D 217/IP 50.

NOTE 2.—This test method is not intended to replace the full-scale penetration as described in Test Method D 217/IP 50. Precision is better in the full-scale penetration method.

5.2 Worked penetration results may be used to establish the consistency of lubricating greases within the above NLGI consistency numbers. The procedures in this test method are widely used for specification purposes, however, no correlation with field performance has been established.

## Standard Test Method for CONE PENETRATION OF LUBRICATING GREASE USING ONE-QUARTER AND ONE-HALF SCALE CONE EQUIPMENT<sup>1</sup>

This standard is issued under the fixed designation D 1403; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers two procedures for measuring the consistency of small samples of lubricating greases by penetration of a  $\frac{1}{4}$ -scale cone or a  $\frac{1}{2}$ -scale cone. This test method includes procedures for the measurement of unworked and worked penetrations.

1.2 Unworked penetrations do not generally represent the consistency of greases in use as effectively as do worked penetrations. The latter are usually preferred for inspecting lubricating greases.

1.3 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1.—In the United States, the equipment dimensions stated in inches are to be regarded as the standard.

## 2. Applicable Document

2.1 *ASTM Standard.*  
D 217 Test for Cone Penetration of Lubricating Grease<sup>2</sup>

2.2 *IP Standard:*

IP 50 Cone Penetration of Lubricating Grease<sup>3</sup>

## 3. Descriptions of Terms Specific to This Standard

3.1 *Penetration of lubricating grease*—the

only one result can be obtained from one filling of a grease cup.

7.2 *Preparing Sample for Measurement*—Place the empty grease worker cup and an appropriate amount of the sample in a container in the water or air bath maintained at  $25^{\circ}\text{C}$  ( $77^{\circ}\text{F}$ ) for sufficient time to bring the temperature of the sample and the worker cup to  $25 \pm 0.5^{\circ}\text{C}$  ( $77 \pm 1^{\circ}\text{F}$ ). Transfer the sample, preferably in one lump, to overfill the cup of the grease worker. Make this transfer in such a manner that the grease will be worked as little as possible. Jar the cup to drive out trapped air and pack the grease with the spatula, with as little manipulation as possible to fill the cup without air pockets. Scrape off the excess grease extending above the rim by moving the blade of the spatula, held inclined toward the direction of motion at an angle of  $45^{\circ}$ , across the rim of the cup. Do not perform any further leveling or smoothing of the surface throughout the determination of unworked penetration and determine the measurement immediately.

7.3 *Cleaning Cone and Shaft*—Clean the penetrometer cone carefully before each test. Bending of the cone shaft can be avoided by holding it securely in its raised position while cleaning. Do not permit grease or oil on the penetrometer shaft, as they can cause drag on the shaft assembly. Do not rotate the cone, as this may cause wear on the release mechanism.

7.4 *Penetration Measurement*—Place the cup on the penetrometer table, making certain that it cannot teeter. Set the mechanism to hold the cone in the "zero" position of the indicator dial, and adjust the apparatus carefully so that the tip of the cone just touches the surface at the center of the test sample. Watching the shadow of the cone tip is an aid to accurate setting. Release the cone shaft rapidly, and allow it to drop for  $5.0 \pm 0.1$  s. The release mechanism should not drag on the shaft. Gently depress the indicator shaft until stopped by the cone shaft and read the penetration to the nearest full unit from the indicator.

7.4.1 If the sample has a penetration over 47 units by  $\frac{1}{4}$  scale or over 97 units by  $\frac{1}{2}$  scale, this sample can then be used for only one test. If the sample has a penetration of 47 units or less by  $\frac{1}{4}$  scale or 97 units or less by  $\frac{1}{2}$  scale, perform three tests in a single cup spacing these tests on three radii  $120^{\circ}$  apart and midway between the center and side of the cup so that the cone will

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<sup>1</sup>This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.G on Lubricating Grease. In the IP, this test method is under the jurisdiction of the Standardization Committee.  
<sup>2</sup>Current edition approved Oct. 28, 1983. Published January 1984. Originally published as D 1403 - 56 T. Last previous edition D 1403 - 69 (1980).  
<sup>3</sup>IP Methods of Analysis and Testing.

neither strike the side of the cup nor impinge on the disturbed area made in a previous test. Center the cone carefully in the container and proceed as in 7.4.

7.5 *Additional Testing*—Make a total of three tests on the sample (either in three cups or in one, as described in 7.4), and report the average value, to the nearest unit, as the unworked penetration of the sample.

#### 8. Procedure for Worked Penetration

8.1 *Sample*—Sufficient sample to overflow the cup of the appropriate grease worker is required.

8.2 *Working*—Transfer sufficient sample to the cup of the clean grease worker to fill it heaping full, mounded up about 7 mm (0.25 in.) at the center, avoiding the inclusion of air by packing with the spatula. Jar the cup from time to time as it is being packed to remove any air inadvertently entrapped. Assemble the worker and, with the vent cock open, depress the plunger to the bottom. Close the vent cock and place the assembled worker in the water or air bath maintained at 25°C (77°F) (Note 5) until the temperature of the worker and contents are at 25 ± 0.5°C (77 ± 1°F). Then remove the worker from the bath and wipe off any water adhering to its surfaces. Subject the grease to 60 full double strokes of the plunger, completed in 1 min ± 5 s, and return the plunger to its top position. Open the vent cock, remove the top and plunger, and return to the cup as much of the grease clinging to the plunger as may readily be removed. As the worked penetration of a lubricating grease may change significantly on standing, proceed in accordance with 8.3, 8.4, and 8.5 immediately.

NOTE 5—If it is desired to immerse the portion of the worker above its closure, take care that the lid is watertight in order to prevent the entrance of water to the worker.

8.3 *Preparing Sample for Measurement*—Prepare the worked sample in the cup for testing so that a uniform and reproducible structure of the grease will be obtained. Jar the cup sharply on the bench or floor and pack the grease down with a spatula to fill the holes left by the plunger and to remove any air pockets (Note 6). Scrape off the excess grease extending above the rim of the cup by moving the blade of the spatula, held inclined toward the direction of motion of an angle of 45°, across the rim of the cup retaining the portion removed (Note 7).

NOTE 6—The jarring should be as vigorous as required to remove the entrapped air without splashing the sample from the cup. In performing these operations, a minimum of manipulation should be used, as any agitation of the grease may have the effect of increasing the working beyond the specified 60 strokes. NOTE 7—Retain the grease removed from the cup in scraping for subsequent tests. Keep the outside of the rim of the cup clean so that the grease forced by the penetrometer cone to overflow the cup may be returned to the cup prior to preparing the sample for the next test.

8.4 *Penetration Measurement*—Determine the penetration of the sample as described in 7.3 and 7.4.

8.5 *Additional Tests*—Immediately make two more tests in succession on the same sample. Return to the cup the portion previously removed with the spatula, then repeat the operations described in 8.3 and 8.4. Report the average of the three tests, to the nearest unit, as the worked penetration of the sample.

#### 9. Calculations

9.1 The penetration values obtained from the  $\frac{1}{4}$ -scale and  $\frac{1}{2}$ -scale cone equipment in 7.5 and 8.5 may be converted to full-scale penetrations (Test Method D 217/IP 50) by the following equations:

9.1.1  $\frac{1}{4}$  Scale:

$$P = 3.75p + 24$$

where:

$P$  = cone penetration by Test Method D 217/IP 50, and

$p$  = cone penetration by  $\frac{1}{4}$ -scale equipment.

NOTE 8—Full-scale penetration values derived from the  $\frac{1}{4}$ -scale conversion equation in 9.1.1 may differ from those obtained using Test Method D 217/IP 50. Parties interested in using full-scale penetration values converted from  $\frac{1}{4}$ -scale results may use a modified conversion equation when mutually agreeable.

9.1.2  $\frac{1}{2}$  Scale:

$$P = 2r + 5$$

where:

$P$  = cone penetration by Test Method D 217/IP 50, and

$r$  = cone penetration by  $\frac{1}{2}$ -scale equipment.

#### 10. Precision

10.1 The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

TABLE 1 Repeatability

	Unworked	Worked	$\frac{1}{4}$ -Scale Units	$\frac{1}{2}$ -Scale Units
	3	3	5	5

TABLE 2 Reproducibility

	Unworked	Worked	$\frac{1}{4}$ -Scale Units	$\frac{1}{2}$ -Scale Units
	10	7	10	13

#### ANNEX

##### (Mandatory Information)

###### A1. APPARATUS

10.1.1 *Repeatability*—The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values in Table 1 in only one case in twenty.

10.1.2 *One-Half Scale Cone and Shaft*—The cone shall be made of steel, stainless steel, or brass with the hardened steel tip of 45 to 50 Hardness, Rockwell C Scale and shall be constructed to conform to the tip of 45 to 50 Hardness, Rockwell C Scale and shall be constructed to conform to the dimensions and tolerances shown in Fig. A1.3. The shaft may be made of stainless steel. The total mass of the cone and its movable attachments shall be  $37.5 \pm 0.050$  g. The mass of the cone shall be  $22.5 \pm 0.025$  g. The mass of the movable attachments shall be  $15 \pm 0.025$  g.

A1.3 *One-Half Scale Cone and Shaft*—The cone

shall be made of plastic or other light-weight material with hardened steel tip of 45 to 50 Hardness, Rockwell C Scale and shall be constructed to conform to the tip of 45 to 50 Hardness, Rockwell C Scale and shall be constructed to conform to the dimensions and tolerances shown in Fig. A1.3. The shaft may be made of stainless steel. The total mass of the cone and its movable attachments shall be  $37.5 \pm 0.050$  g. The mass of the cone shall be  $22.5 \pm 0.025$  g. The mass of the movable attachments shall be  $15 \pm 0.025$  g.

A1.4 *One-Half Scale Grease Worker*—Worker shall conform to the dimensions given in Fig. A1.4. Other methods of fastening the cover and securing the worker may be used. The worker may be constructed for either manual or mechanical operation. Design shall be such that a rate of  $60 \pm 10$  strokes per minute, with a minimum length of  $\frac{1}{16}$  in. (14 mm), can be maintained.

A1.1 *One-Quarter Scale Cone and Shaft*—The cone shall be made of plastic or other light-weight material with hardened steel tip of 45 to 50 Hardness, Rockwell C Scale and shall be constructed to conform to the dimensions and tolerances shown in Fig. A1.1. The shaft may be made of magnesium. The total mass of the cone and its movable attachments shall be  $9.38 \pm 0.025$  g. The total mass of the cone and its movable attachments may be adjusted by adding small shot to the cavity of the shaft.

A1.2 *One-Quarter Scale Grease Worker*—Worker shall conform to the dimensions given in Fig. A1.2. Other methods of fastening the cover and securing the worker may be used. The worker may be constructed for either manual or mechanical operation. Design shall be such that a rate of  $60 \pm 10$  strokes per minute, with a minimum length of  $\frac{1}{16}$  in. (35 mm), can be maintained.

9.2 *Additional Tests*—Immediately make two more tests in succession on the same sample. Return to the cup the portion previously removed with the spatula, then repeat the operations described in 8.3 and 8.4. Report the average of the three tests, to the nearest unit, as the worked penetration of the sample.

NOTE 9—Full-scale penetration values derived from the  $\frac{1}{4}$ -scale conversion equation in 9.1.1 may differ from those obtained using Test Method D 217/IP 50. Parties interested in using full-scale penetration values converted from  $\frac{1}{4}$ -scale results may use a modified conversion equation when mutually agreeable.

NOTE 10—If it is desired to immerse the portion of the worker above its closure, take care that the lid is watertight in order to prevent the entrance of water to the worker.

9.3 *Preparing Sample for Measurement*—Prepare the worked sample in the cup for testing so that a uniform and reproducible structure of the grease will be obtained. Jar the cup sharply on the bench or floor and pack the grease down with a spatula to fill the holes left by the plunger and to remove any air pockets (Note 6). Scrape off the excess grease extending above the rim of the cup by moving the blade of the spatula, held inclined toward the direction of motion of an angle of 45°, across the rim of the cup retaining the portion removed (Note 7).

9.4 *Penetration Measurement*—Determine the penetration of the sample as described in 7.3 and 7.4.

9.5 *Additional Tests*—Immediately make two more tests in succession on the same sample. Return to the cup the portion previously removed with the spatula, then repeat the operations described in 8.3 and 8.4. Report the average of the three tests, to the nearest unit, as the worked penetration of the sample.

NOTE 11—Full-scale penetration values derived from the  $\frac{1}{4}$ -scale conversion equation in 9.1.1 may differ from those obtained using Test Method D 217/IP 50. Parties interested in using full-scale penetration values converted from  $\frac{1}{4}$ -scale results may use a modified conversion equation when mutually agreeable.

NOTE 12—If it is desired to immerse the portion of the worker above its closure, take care that the lid is watertight in order to prevent the entrance of water to the worker.

9.6 *Calculations*

9.6.1 *One-Quarter Scale Cone and Shaft*—The cone

shall be made of plastic or other light-weight material with hardened steel tip of 45 to 50 Hardness, Rockwell C Scale and shall be constructed to conform to the dimensions and tolerances shown in Fig. A1.1. The shaft may be made of magnesium. The total mass of the cone and its movable attachments shall be  $9.38 \pm 0.025$  g. The total mass of the cone and its movable attachments may be adjusted by adding small shot to the cavity of the shaft.

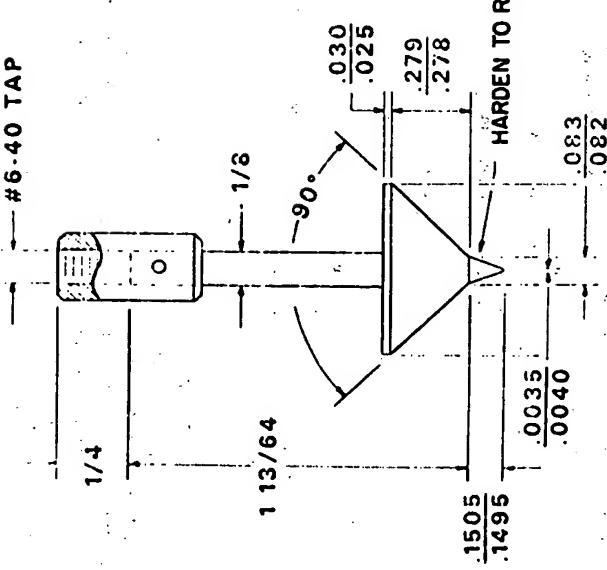
9.6.2 *One-Quarter Scale Grease Worker*—Worker shall conform to the dimensions given in Fig. A1.2. Other methods of fastening the cover and securing the worker may be used. The worker may be constructed for either manual or mechanical operation. Design shall be such that a rate of  $60 \pm 10$  strokes per minute, with a minimum length of  $\frac{1}{16}$  in. (35 mm), can be maintained.

9.7 *Repeatability*—The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values in Table 1 in only one case in twenty.

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All dimensions

**TOTAL MASS OF CONE AND  
ITS MOVABLE ATTACHMENTS**  
 **$= 9.38 \pm 0.0259$**

Inches	Millimetres
$\frac{1}{4}$	3.5
$\frac{1}{8}$	6.4
$\frac{13}{64}$	30.5
0.0035	0.09
0.0040	0.10
0.025	0.63
0.030	0.76
0.082	2.08
0.083	2.11
0.1495	3.8
0.1505	3.82
0.278	7.06
0.279	7.09
0.639	16.23
0.640	16.26

EIG A1.1 One Quantitative Sample Case

DETAILS OF GREASE PLATE.

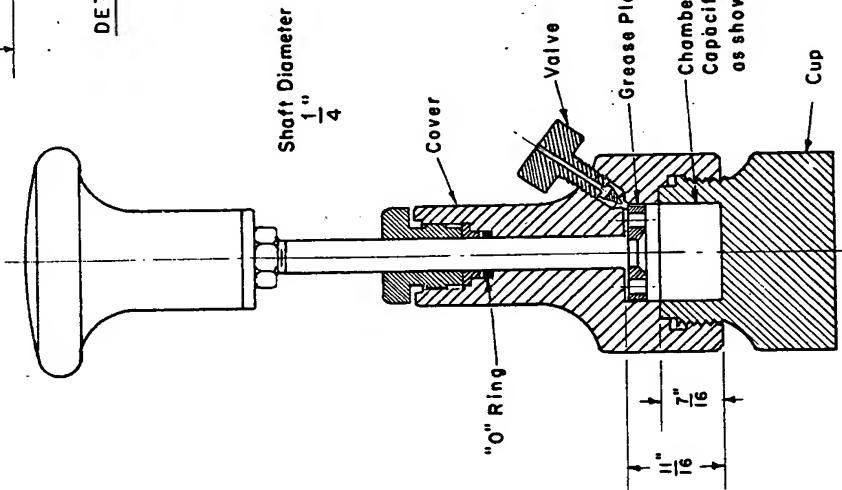


FIG. A1.3 One-quarter Scale Grease Worker

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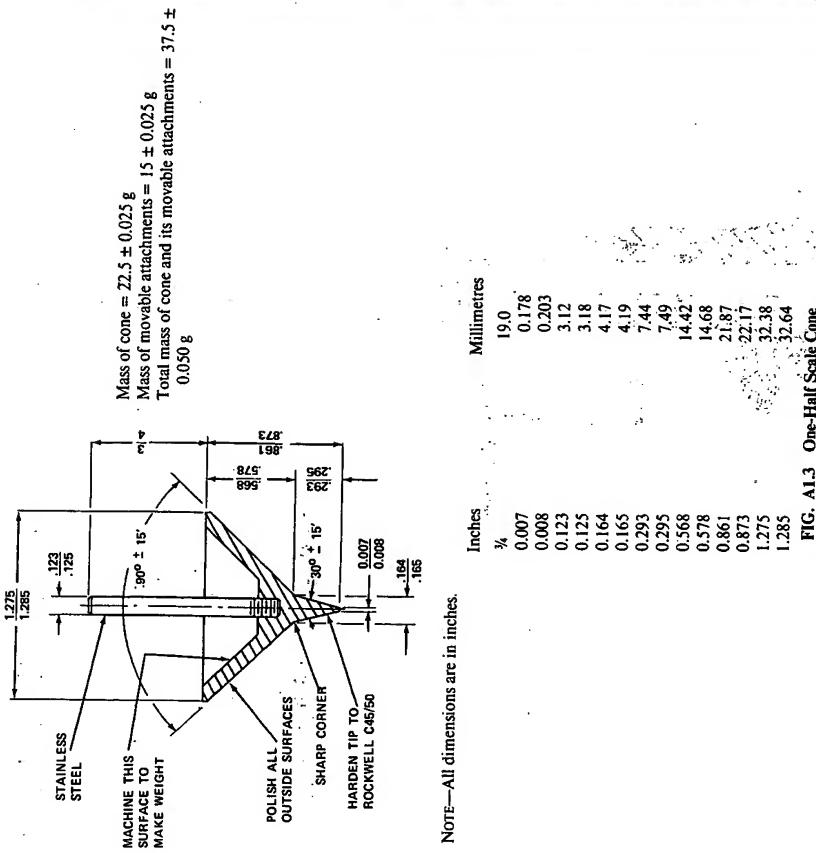
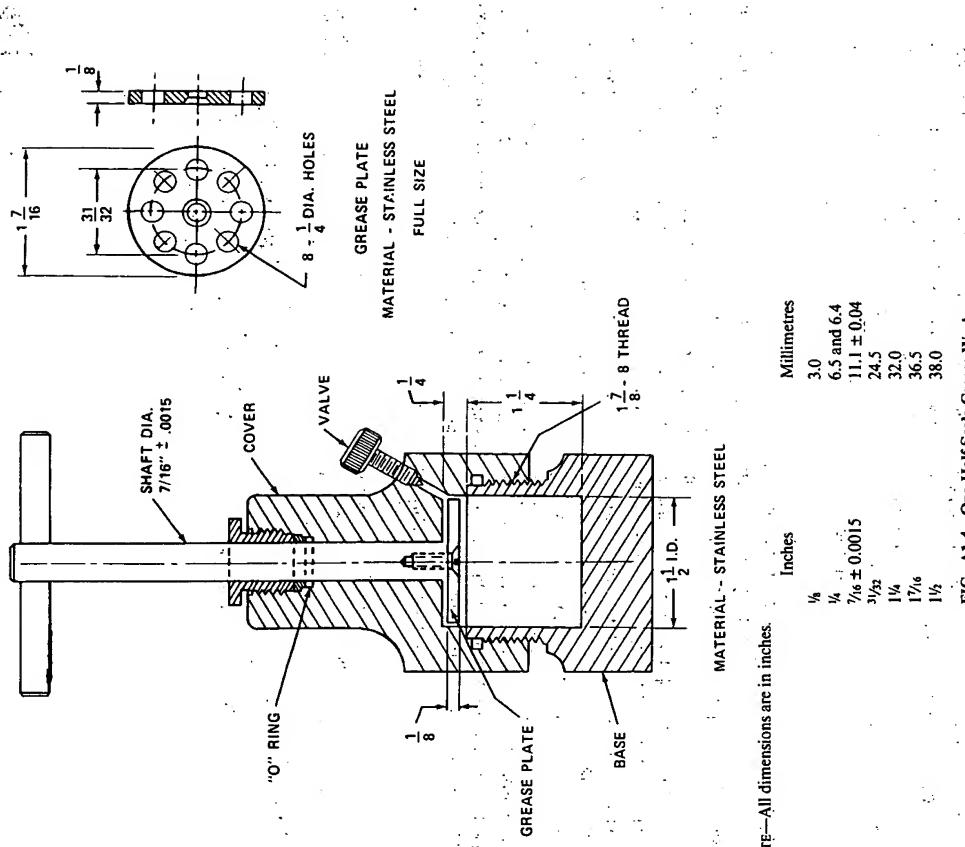


FIG. A1.3 One Half Scale Cross

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